N62661.AR.002888
NS NEWPORT
5090.3a

FINAL SAMPLING AND ANALYSIS PLAN ADDENDUM FOR CODDINGTON COVE RUBBLE FILL AREA NS NEWPORT RI

11/12/2013

RESOLUTION CONSULTANTS

SAMPLING AND ANALYSIS PLAN ADDENDUM
Coddington Cove Rubble Fill (CCRF) Area, Naval Station (NAVSTA) Newport, Rhode Island

FINAL

Prepared for:

Department of the Navy
Naval Facilities Engineering Command, Mid-Atlantic
9742 Maryland Ave.
Norfolk, VA 23511-3095

Contract Number N62470-11-D-8013

CTO WE19

Prepared by:

Resolution Consultants
A Joint Venture of AECOM & EnSafe
1500 Wells Fargo Building
440 Monticello Avenue
Norfolk, VA 23510

November 12, 2013

SAP Worksheet #1: Title and Approval Page

Final

SAMPLING AND ANALYSIS PLAN ADDENDUM

November 12, 2013

Coddington Cove Rubble Fill (CCRF) Area
NAVSTA, Newport, RI

Prepared for:
Department of the Navy
Naval Facilities Engineering Command, Mid-Atlantic
9742 Maryland Ave.
Norfolk, VA 23511-3095

Prepared by:
Resolution Consultants
A Joint Venture of AECOM & EnSafe
1500 Wells Fargo Building
440 Monticello Avenue
Norfolk, VA 23510

Prepared under:
Contract Number N62470-11-D-8013

CTO WE19

Review Signature: Cannon, Melissa — Digitally signed by Cannon, Melissa; DN: cn=Cannon, Melissa, ou=USSGM1; Date: 2013.11.07 10:44:36 -05'00'

Melissa Cannon, CTO Manager — Date
Resolution Consultants

Approval Signature:

Monique Nixon/Chemist, QA Manager — Date
US Navy

Other Approval Signature: Maritza Montegross — Digitally signed by MONTEGROSS.MARITZA.L.1025648036; DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=MONTEGROSS.MARITZA.L.1025648036; Date: 2013.11.07 16:34:32 -05'00'

Maritza Montegross, Navy RPM — Date
US Navy

SAP Worksheet #1: Title and Approval Page

Draft

SAMPLING AND ANALYSIS PLAN ADDENDUM

May 13, 2013

Coddington Cove Rubble Fill (CCRF) Area
NAVSTA, Newport, RI

Prepared for:
Department of the Navy
Naval Facilities Engineering Command, Mid-Atlantic
9742 Maryland Ave.
Norfolk, VA 23511-3095

Prepared by:
Resolution Consultants
A Joint Venture of AECOM & EnSafe
1500 Wells Fargo Building
440 Monticello Avenue
Norfolk, VA 23510

Prepared under:
Contract Number N62470-11-D-8013

CTO WE19

Review Signature:	Cannon, Melissa — Digitally signed by Cannon, Melissa, DN: cn=Cannon, Melissa, ou=USSGM1, Date: 2013.05.13 10:53:45 -04'00'	
	Melissa Cannon, CTO Manager Resolution Consultants	Date
Approval Signature:	NIXON.MONIQUE.L.1272150628 — Digitally signed by NIXON.MONIQUE.L.1272150628, DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=NIXON.MONIQUE.L.1272150628, Date: 2013.05.13 12:57:17 -04'00'	
	Monique Nixon/Chemist, QA Manager US Navy	Date
Other Approval Signature:	MONTEGROSS.MARITZA.L.1025648036 — Digitally signed by MONTEGROSS.MARITZA.L.1025648036, DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=MONTEGROSS.MARITZA.L.1025648036, Date: 2013.05.13 12:34:59 -04'00'	
	Maritza Montegross, Navy RPM US Navy	Date

EXECUTIVE SUMMARY

This Work Plan has been prepared in the form of a Sampling and Analysis Plan (SAP) Addendum by Resolution Consultants (Resolution) for the U.S. Department of the Navy (Navy) and the Naval Facilities Engineering Command (NAVFAC). This SAP pertains to monitoring well installation and additional groundwater sampling at the Coddington Cove Rubble Fill (CCRF) Area (Site 4) at Naval Station (NAVSTA) Newport, Rhode Island and is an addendum to Tetra Tech's Final SAP dated November 2010 (Appendix A). A Draft Study Area Screening Evaluation (SASE) Report was drafted and five metals in groundwater were identified as posing potential risks:

- Arsenic (As);
- Chromium (Cr);
- Cobalt (Co);
- Iron (Fe); and
- Manganese (Mn).

Following the installation of two upgradient monitoring wells, an additional round of groundwater sampling will provide more data to confirm concentrations of these metals in groundwater, and to evaluate whether they are site-related. In addition to analyzing for these metals, additional geochemical and biological parameters will be collected to evaluate whether natural subsurface conditions are causing the increased presence of these metals in groundwater.

The primary objective of the planned investigation is to provide additional data necessary to fill the groundwater data gaps, to complete the SASE, and collect data that may be necessary if results indicate that potential risks do exist and a feasibility study (FS) is warranted. Analytical parameters to achieve the specific goals listed above are summarized in Table ES-1.

Table ES-1
Analytical Parameters Designed to Achieve Investigation Goals
CCRF, NAVSTA Newport, RI

Analytical Parameter	Matrix
Parameters of Interest	
Metals (As, Cr, Co, Fe, Mn)	GW
Hexavalent chromium (Cr^{+6})	GW
Geochemical and Biological Indicator Data	
TOC	GW
TDS	GW
Alkalinity (bicarbonate and carbonate)	GW
Chloride, nitrite/nitrate, sulfate	GW
Ammonia	GW
Metals (Al, Ca, Mg, K, Na)	GW

Notes:
GW – Groundwater
TAL – Target Analyte List

CONTENTS

SAP Worksheet #1: Title and Approval Page ..... WS 1-1
SAP Worksheet #2: Sampling and Analysis Plan Identifying Information ..... WS 2-1
SAP Worksheet #3: Distribution List ..... WS 3-1
SAP Worksheet #4: Project Personnel Sign-Off Sheet ..... WS 4-1
SAP Worksheet #5: Project Organizational Chart ..... WS 5-1
SAP Worksheet #6: Communication Pathways ..... WS 6-1
SAP Worksheet #7: Personnel Responsibilities Table ..... WS 7-1
SAP Worksheet #8: Special Personnel Training Requirements Table ..... WS 8-1
SAP Worksheet #9: Project Scoping Session Participants Sheet ..... WS 9-1
SAP Worksheet #10: Conceptual Site Model ..... WS 10-1
SAP Worksheet #11: Project Quality Objectives/Systematic Planning Process Statements ..... WS 11-1
SAP Worksheet #12 Measurement Performance Criteria – Field QC Samples ..... WS 12-1
SAP Worksheet #13: Secondary Data Criteria and Limitations Table ..... WS 13-1
SAP Worksheet #14: Summary of Project Tasks ..... WS 14-1
SAP Worksheet #15: Establishing Laboratory Reference Limits ..... WS 15-1
SAP Worksheet #16: Project Schedule/Timeline Table ..... WS 16-1
SAP Worksheet #17: Sampling Design and Rationale ..... WS 17-1
SAP Worksheet #18: Location-Specific Sampling Methods/SOP Requirements Table ..... WS 18-1
SAP Worksheet #19: Field Sampling Requirements Table[1] ..... WS 19-1
SAP Worksheet #20: Field Quality Control Sample Summary Table ..... WS 20-1
SAP Worksheet #21: Project Sampling SOP References Table ..... WS 21-1
SAP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table ..... WS 22-1
SAP Worksheet #23: Analytical SOP References Table[1] ..... WS 23-1
SAP Worksheet #24: Analytical Instrument Calibration Table ..... WS 24-1
SAP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table ..... WS 25-1
SAP Worksheet #26: Sample Handling System ..... WS 26-1
SAP Worksheet #27: Sample Custody Requirements ..... WS 27-1
SAP Worksheet #28: Laboratory QC Samples Table ..... WS 28-1
SAP Worksheet #29: Project Documents and Records Table ..... WS 29-1
SAP Worksheet #30: Analytical Services Table ..... WS 30-1
SAP Worksheet #31: Planned Project Assessments Table ..... WS 31-1
SAP Worksheet #32: Assessment Findings and Corrective Action Responses Table ..... WS 32-1
SAP Worksheet #33: Quality Assurance Management Reports Table ..... WS 33-1
SAP Worksheets #34-36: Data Verification and Validation (Steps I and IIa/IIb) Process Table ..... WS 34-36-1
SAP Worksheet #37: Usability Assessment ..... WS 37-1
References R-1

Figures

Figure 1 Regional Location, Coddington Cove Rubble Fill (CCRF) Area
Figure 2 Site Plan, Groundwater Monitoring Results (Metals), and Proposed Monitoring Well Locations
Figure 3 Geologic Cross Section Location Plan, Section A-A' and B-B'
Figure 4 Cross Sections A-A' and B-B'

Appendices

(Refer to CD Provided with Document)

Appendix A November 2010 Final Sampling and Analysis Plan
Appendix B Resolution Consultants SOPs
Appendix C Laboratory Certifications
Appendix D Topographic Map and Aerial Photographs

ACRONYMS AND ABBREVIATIONS

°C	Degrees Celsius
BSG	Below Surface Grade
CCRF	Coddington Cove Rubble Fill
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLEAN	Comprehensive Long-Term Environmental Action, Navy
CoC	Chain-of-Custody
CSM	Conceptual Site Model
CTO	Contract Task Order
DL	Detection Limit
DO	Dissolved Oxygen
DQI	Data Quality Indicator
ELAP	Environmental Laboratory Accreditation Program
FTL	Field Team Leader
FTMR	Field Task Modification Request
IDW	Investigation-Derived Waste
LOD	Limit of Detection
LOQ	Limit of Quantitation
MCL	Maximum Contaminant Level
MS/MSD	Matrix Spike/Matrix Spike Duplicate
N/A	Not Applicable
NAVFAC MIDLANT	Naval Facilities Engineering Command, Mid-Atlantic
NAVSTA	Naval Station
Navy	U.S. Department of the Navy
NEDD	Navy Electronic Data Deliverable
NIRIS	Naval Installation Restoration Information Solution
NTR	Navy Technical Representative
NTU	Nephelometric Turbidity Unit
ORP	Oxidation-Reduction Potential
OU	Operable Unit
PAL	Project Action Limit
PDF	Portable Document Format
PM	Project Manager
POC	Point of Contact
PQOs	Project Quality Objectives
QA/QC	Quality Assurance/Quality Control
QSM	Quality Systems Manual
RAOs	Remedial Action Objectives
Resolution	Resolution Consultants
RIDEM	Rhode Island Department of Environmental Management
RPD	Relative Percent Difference
RPM	Remedial Program Manager
RTC	Response to Comments
RSL	Regional Screening Levels

SAP	Sampling and Analysis Plan
SASE	Study Area Screening Evaluation
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SSO	Site Safety Officer
TAL	Target Analyte List
TAT	Turnaround Time
TBD	To Be Determined
TCL	Target Compound List
TOC	Total Organic Carbon
TDS	Total Dissolved Solids
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
USEPA	United States Environmental Protection Agency

SAP Worksheet #2: Sampling and Analysis Plan Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: Coddington Cove Rubble Fill (CCRF) Area, Site 4

Operable Unit: Site 4 (No OU designation)

Contractor Name: Resolution Consultants (Resolution)

Contract Number: N62470-11-D-8013

Contract Title: Atlantic Comprehensive Long-Term Environmental Action, Navy (CLEAN), Naval Facilities Engineering Command, Mid-Atlantic (NAVFAC MIDLANT)

Work Assignment Number: Contract Task Order (CTO) WE-19

1. This Sampling and Analysis Plan (SAP) Addendum was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (USEPA, 2005) and United States Environmental Protection Agency (USEPA) *Guidance for Quality Assurance Project Plans, USEPA QA/G-5* (USEPA, 2002).

2. Regulatory program: Installation Restoration (IR) Program, which follows the general framework of Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), with consideration of Rhode Island Department of Environmental Management (RIDEM) review and input.

3. This SAP is a Tier I project-specific SAP Addendum for CCRF. This SAP is an addendum to Tetra Tech's November 2010 Final SAP (Appendix A).

4. List organizational partners (stakeholders) and identify the connection with lead organization:

Organization Partners/Stakeholders	Connection
Navy, NAVFAC MIDLANT	Responsible Federal Agency
USEPA, Region 1	Federal Regulatory Agency
RIDEM	State Regulatory Agency

5. Lead organization: NAVFAC MIDLANT

6. If any required SAP elements and required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below: N/A

SAP Worksheet #3: Distribution List

(UFP-QAPP Manual Section 2.3.1)

SAP Recipients	Title	Organization	Telephone Number (optional)	E-mail Address or Mailing Address
Winoma Johnson	Northeast Technical Leader	NAVFAC MIDLANT	757.341.2008	Winoma.Johnson@navy.mil
Maritza Montegross	Remedial Project Manager (RPM)	NAVFAC MIDLANT	757.341.2013	Maritza.Montegross@navy.mil
Darlene Ward	NAVSTA Newport CCRF Site POC	NAVFAC MIDLANT	401.841.6376	Darlene.Ward@navy.mil
David Dorocz	NAVSTA Newport Facility POC	NAVFAC MIDLANT	401.841.7671	David.Dorocz@navy.mil
Lynne Jennings	Federal Facilities Section Chief	USEPA, Region 1	617.918.1210	Jennings.Lynne@epa.gov
Bill Lovely	RPM	USEPA, Region 1	617.918.1240	Lovely.william@epa.gov
Gary Jablonski	Principal Sanitary Engineer	RIDEM	401.222.2797 ext. 7148	Gary.Jablonski@dem.ri.gov
Pamela Crump	Sanitary Engineer, RPM	RIDEM	401.222.2797 ext. 7020	Pamela.Crump@dem.ri.gov
Mark Kauffman	NAVSTA Newport Activity Coordinator	Resolution	978.905.2262	Mark.Kauffman@aecom.com
Melissa Cannon	CTO Manager	Resolution	978.400.1213	Melissa.Cannon@aecom.com
Waverly Braunstein	Project Chemist/Data Validation Manager	Resolution	978.905.2133	Waverly.Braunstein@aecom.com
Elizabeth Perry	Hydrogeologist	Resolution	978.905.2359	Elizabeth.Perry@aecom.com
Josh Millard	Field Team Leader/Site Safety Officer	Resolution	978.905.2324	Joshua.Millard@aecom.com
Jennifer Obrin	Analytical Laboratory Manager	Katahdin Analytical Services, Inc.	207.874.2400	jobrin@katahdinlab.com
Glen Wagner	NIRIS/Administrative Record Coordinator	Tetra Tech	412.320.2211	Niris-support@tetratech.com

Notes:
Distribution of the SAP can include a copy on the submittal cover letter, a hardcopy, an electronic copy via CD or email and/or an upload via NIRIS. Distribution may vary depending on the deliverable version (e.g., draft, draft final, final) and/or individual preferences of SAP recipients.

SAP Worksheet #4: Project Personnel Sign-Off Sheet

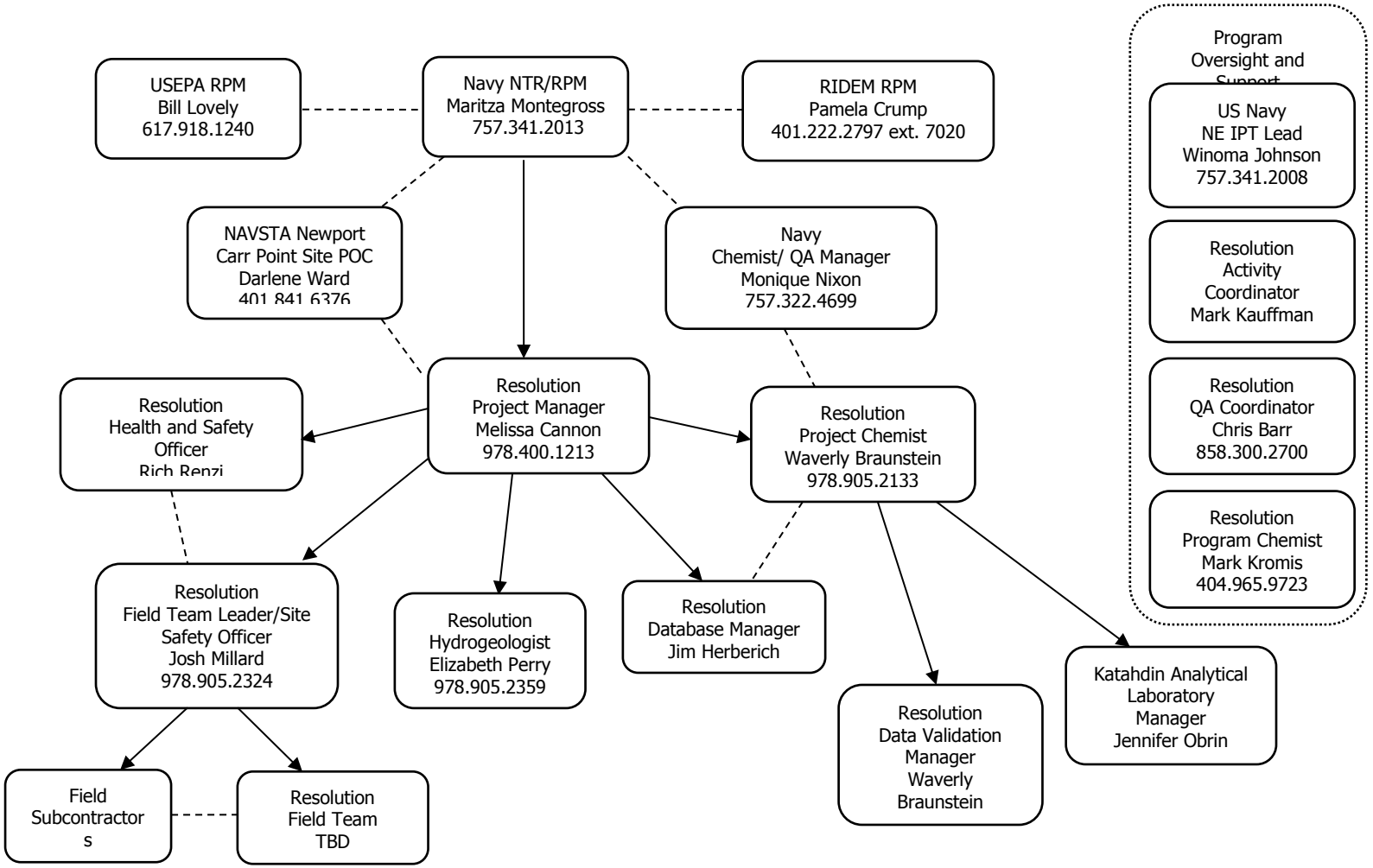
(UFP-QAPP Manual Section 2.3.2)

All key personnel are to receive a copy of this project-specific SAP and will be required to read and understand the SAP prior to performing project tasks. The following table will be modified as applicable and used to document this. This table as well as worksheet #1 will be completed and maintained within Resolution's files.

Project Personnel	Title/Organization	Telephone Number	Signature	Date
Maritza Montegross	Navy Technical Representative (NTR)/ Remedial Project Manager (RPM), NAVFAC MIDLANT	757.341.2013		
Mark Kauffman	Activity Coordinator, Resolution	978.905.2262		
Melissa Cannon	CTO Manager, Resolution	978.400.1213		
Waverly Braunstein	Project Chemist/ Data Validation Manager, Resolution	978.905.2133		
Elizabeth Perry	Hydrogeologist	978.905.2359		
Josh Millard	Field Team Leader/ Site Safety Officer, Resolution	978.905.2324		
Jennifer Obrin	Analytical Laboratory Manager, Katahdin Analytical Services	207.874.2400		

SAP Worksheet #5: Project Organizational Chart

Lines of Authority ———— Lines of Communication - - - - - - - - - - - -



SAP Worksheet #6: Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

The communication pathways for the SAP are shown below.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Regulatory Agency Interface	NAVFAC MIDLANT	Maritza Montegross	757.341.2013	Navy RPM will interface with Regulatory Agency directly via phone or email as needed.
Field Progress Reports	Resolution	Melissa Cannon	978.400.1213	The Resolution Field Team Leader will email daily field progress reports to the Resolution PM. In addition, there will be phone conversation between the Resolution Field Team Leader and the Resolution PM as needed.
Stop Work due to Safety Issues	Resolution	SSO – Josh Millard	978.905.2324	The Resolution SSO will verbally inform onsite personnel, including subcontractors as soon as possible. The Resolution SSO will verbally inform the Resolution PM of the Stop Work condition as well as when it is resolved. As need be, the Resolution SSO and/or PM will work with the Resolution Health & Safety Officer to resolve issues.
SAP/WP Changes prior to Field/ Laboratory Work	Resolution	Melissa Cannon	978.400.1213	The Resolution PM will notify the Navy RPM either verbally or via email of any planned amendments to the SAP. The Resolution PM will document the changes via a Field Task Modification Request (FTMR) form and a concurrence letter.
SAP/WP Changes in the Field	Resolution	FTL – Josh Millard	978.905.2324	The Resolution Field Team Leader will verbally notify the Resolution PM as soon as practical of realizing a need for an amendment. The Resolution PM will notify the Navy RPM either verbally or via email the same day of the realized change and the changes will be documented in a scheduled impact letter to the Navy RPM.
Field Corrective Actions	Resolution	FTL – Josh Millard	978.905.2324	The need for corrective action for field issues will be determined by the Resolution Field Team Leader. The Resolution Field Team Leader will notify the Resolution PM.
Sample Receipt Variances	Resolution	Waverly Braunstein	978.905.2133	The laboratory will notify the Resolution Project Chemist of any issues with respect to sample receipt. The Resolution Project Chemist will notify the Resolution PM and/or Field Team Leader.
Reporting Lab Quality Variances	Resolution	Waverly Braunstein	978.905.2133	The laboratory will notify the Resolution Project Chemist of any issues in the analytical report case narrative.
Analytical Corrective Actions	Resolution	Waverly Braunstein	978.905.2133	The need for corrective action for analytical issues will be determined by the Resolution Project Chemist.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Laboratory Quality Issues	Resolution	Waverly Braunstein	978.905.2133	The Resolution Project Chemist will make the Resolution PM aware of any laboratory quality issues. The Resolution PM will notify the Navy RPM if any laboratory quality issues will significantly impact the project (i.e., significant meaning data is not useable or project schedule or costs have been majorly impacted). As appropriate, the Navy RPM will notify the Navy Chemist to ensure other Navy projects are not impacted.
Reporting Data Validation Issues	Resolution	Waverly Braunstein	978.905.2133	The Resolution Project Chemist will provide the Resolution PM with a report of any issues affecting data quality as identified during the validation process. As necessary, the Resolution Project Chemist will notify the laboratory as soon as possible if issues are found with the data and work with the laboratory to resolve any issues.
Data Validation Corrective Actions	Resolution	Waverly Braunstein	978.905.2133	The Resolution Project Chemist will notify the Resolution PM of any actions that may be required as a result of the data validation.

SAP Worksheet #7: Personnel Responsibilities Table

(UFP-QAPP Manual Section 2.4.3)

Title/Role	Organizational Affiliation	Responsibilities
Maritza Montegross, RPM	NAVFAC MIDLANT	Responsible for coordination/oversight of IR activities at CCRF.
Mark Kauffman, Activity Coordinator	Resolution	Provides senior oversight and review. Coordination among NAVSTA Newport sites.
Melissa Cannon, CTO Manager	Resolution	Responsible for coordination/oversight of investigation activities associated with this SAP.
Waverly Braunstein, Project Chemist	Resolution	Responsible for oversight of/coordination with laboratory and data validators and management of sample tracking.
Rich Renzi, H&S Officer	Resolution	Responsible for oversight of health and safety for field activities.
Josh Millard, FTL/SSO	Resolution	Responsible for coordination of field activities; supervises field events; ensures the implementation of the health and safety plan.
Jennifer Obrin, Laboratory PM	Katahdin Analytical Services	Responsible for coordination/oversight of analytical services.
Elizabeth Perry, Hydrogeologist	Resolution	Responsible for oversight/coordination of data evaluation/interpretation.

SAP Worksheet #8: Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

There are no special personnel training requirements related to this SAP Addendum.

SAP Worksheet #9: Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name:	CCRF SAP Addendum	**Site Name:**	CCRF
Projected Date(s) of Sampling:	December 2013	**Site Location:**	CCRF, NAVSTA, Newport, RI
Project Manager:	Melissa Cannon		
Date of Session:	1/29/2013		
Scoping Session Purpose:	Partnering Team Meeting		

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Maritza Montegross	NTR/RPM	NAVFAC MIDLANT	757.341.2013	Maritza.Montegross@navy.mil	Coordinates IR Activities
Dave Barclift	Navy Risk Assessor	NAVFAC LANT	215.897.4913	David.Barclift@navy.mil	Risk Assessor
Tom Campbell	Former Project Manager	Tetra Tech	978.474.8400	Thomas.Campbell@tetratech.com	Former Contract Project Manager
Mark Kauffman	Activity Coordination	Resolution	978.905.2262	Mark.Kauffman@aecom.com	Senior Oversight/Review
Melissa Cannon	CTO Manager	Resolution	978.400.1213	Melissa.Cannon@aecom.com	Oversight of SAP Production

Comments/Decisions:

- Discussed the site history, specifically previous work conducted as part of the SASE.
- Discussed the need for additional investigation (i.e., results of the investigation completed to date as part of the SASE).
- Discussed the scope of the additional investigation.
- Discussed how to proceed in regards to the work plan for the additional investigation.

Action Items:

- Tetra Tech to provide Resolution with necessary documents.
- Resolution to provide Navy with concurrence letter to prepare a SAP Addendum.

Consensus Decisions:

- Resolution will prepare a SAP Addendum to include:
    - Installation of an off-site, upgradient monitoring well; and
    - Additional groundwater sampling of on-site monitoring wells for select metals and geochemical/biological indicator data.
- Following the additional field investigation, Resolution will complete research, data evaluation and preparation of a Technical Memorandum (Tech Memo) to discuss the results of the additional groundwater sampling (including upgradient groundwater quality) and the geochemical evaluation.
- The Tech Memo will be provided to Tetra Tech and Tetra Tech will append to the SASE and finalized the SASE incorporating the finding documented in the tech memo.

SAP Worksheet #10: Conceptual Site Model

(UFP-QAPP Manual Section 2.5.2)

Overview

The Coddington Cove Rubble Fill (CCRF) Area (Site 4) is a former fill/disposal area and wetland located at Naval Station (NAVSTA) Newport. The site, an approximate 8-acre vacant lot, is situated on the northwest side of Coddington Highway and is located in Middletown and Newport, Rhode Island. The boundary between Newport and Middletown crosses through the site in a northwest-southeast direction. Refer to Figure 1 for the regional location of the site. Please refer to Worksheet 10 of the parent SAP (Tetra Tech, November 2010), included as Appendix A, for additional details regarding the Conceptual Site Model (CSM).

A Draft Study Area Screening Evaluation (SASE) Report was drafted and five metals in groundwater were identified as posing potential risks:

- Arsenic (As);
- Chromium (Cr);
- Cobalt (Co);
- Iron (Fe); and
- Manganese (Mn).

Analytical results for these metals in groundwater are summarized on Table 10-1, Figure 2, and Figure 4. For preliminary comparison purposes to refine the details of this SAP Addendum, prior analytical results are compared to initial screening values listed on Table 10-1. The initial screening values applied consists of the USEPA maximum contaminant levels (MCLs), from the USEPA Drinking Water Standards and Health Advisories, May 2009, where listed. For the metals with no listed MCL, the initial screening values applied consist of the USEPA Regional Screening Levels (RSLs) for Tapwater, May 2010. The RSL values were adjusted for a target hazard quotient of 0.1 to account for cumulative effects on the same target organ.

In general, the highest concentrations of these metals are encountered in two areas of the site, which include the north central area at wells MW-04, MW-07, and MW-08, and the southwestern site boundary at MW-01 and MW-02. Iron is highest at MW-07 and MW-08, while manganese is highest at MW-1 and MW-4. Based on the Draft SASE, the potential risks associated with these metals in groundwater are for the hypothetical future resident using groundwater as a source of drinking water. However, the exposure point concentration (EPC) for arsenic used in the risk assessment was 6.4 ug/l, which is below the MCL of 10 ug/l. Therefore, the groundwater meets drinking water standards for arsenic. The EPC for chromium was less than 1 ug/l, and while this is

above the initial screening value for hexavalent chromium, it is well below the screening level for trivalent chromium. The relatively high concentrations of iron and manganese indicate an anaerobic (low in oxygen) groundwater system. Under these geochemical conditions, trivalent chromium would be the expected species present, rather than the oxidized form of hexavalent chromium. Anaerobic conditions leading to elevated iron and manganese are common in many groundwater systems, and the cobalt may be associated with iron and/or manganese oxides. As shown on the cross-sections in Figure 4, anaerobic conditions characterize the majority of the groundwater system across the site as indicated by dissolved oxygen (DO) concentrations below 1 mg/L, and negative oxidation reduction potential (ORP) values. As shown at MW-08 and MW-07 on cross-section B-B', the lowest DO concentrations (0.44 mg/L and 0.54 mg/L) and low ORP values (-107.8 mV and -89.6 mV) correspond to the highest iron concentrations 78,000 and 32,000 µg/L (Figure 4).

Figure 3 presents the locations of geologic cross-sections A-A' and B-B', and the geologic cross-sections are presented on Figure 4. The cross-sections are oriented perpendicular and parallel to groundwater flow direction, respectively. Included on the cross-sections are stratigraphic contacts for fill, sand and gravel, and weathered bedrock, as well as the water table elevation. The well screens, also shown, are located primarily within the sand and gravel unit below the fill, although a few are also partially screened in the fill, and some are fully saturated while others bracket the water table. Also shown are analytical results for cobalt, iron, and manganese in soil and water, total petroleum hydrocarbons (TPH) in soil, summation of VOCs and SVOCs in water, and field screening results for DO, ORP, pH, and specific conductance in groundwater. Concentrations of TPH were consistently detected in the upper ten feet of soil across the site (with the exception of MW-4 and MW-6) and within the well screened interval. Total metals concentrations in soil for cobalt, iron, and manganese show little variation vertically or horizontally across the site. Although TPH in soil is below detection limits at MW-04, the total VOC and SVOC concentration is high. Petroleum like odor was reported in soil at MW-01. The results indicate the presence of organics in soil and groundwater, and a reducing groundwater system.

An additional round of groundwater sampling will provide more data to confirm concentrations of the metals in groundwater, and to evaluate whether they are site-related. In addition to analyzing for these metals, supplemental geochemical and biological parameters will be measured to evaluate whether natural subsurface conditions are causing the increased presence of these metals in groundwater. Also, new groundwater monitoring wells will be installation to provide an additional data point upgradient of the site.

Elevated concentrations of metals in groundwater could be attributable to naturally-occurring reducing conditions in the subsurface soil. The solubility of these metals could be increased by several possible causes. Low oxygen conditions, common in wetland areas, convert metals into their soluble forms, such as Fe^{+3} to Fe^{+2}. In addition, most Cr is typically present as Cr^{+3}, particularly in reduced, low-oxygen conditions.

The primary objective of the planned investigation is to provide additional data necessary to fill the groundwater data gaps, assess possible factors that may influence groundwater concentrations, and to complete the SASE.

TABLE 10-1
Summary of Target Metals in Groundwater
Coddington Cove Rubble Fill Area (IR Site 4)
NAVSTA Newport, Newport RI

Sampling Location					**MW-01**	**MW-02**	**MW-03**	**MW-04**	**MW-05**	**MW-06**	**MW-06**	**MW-07**	**MW-08**
Sampling Date	**Initial Screening Value (µg/L)**				12/21/2010	12/21/2010	12/22/2010	12/21/2010	12/21/2010	12/20/2010	12/20/2010	12/20/2010	12/20/2010
Sample Type					N	N	N	N	N	N	FD	N	N
Sample ID		**Minimum**	**Maximum**	**Average**	CRF-GW-MW01-1210	CRF-GW-MW02-1210	CRF-GW-MW03-1210	CRF-GW-MW04-1210	CRF-GW-MW05-1210	CRF-GW-MW06-1210	CRF-GW-MW06-1210-D	CRF-GW-MW07-1210	CRF-GW-MW08-1210
Dissolved Metals (µg/L)													
Arsenic	10[a]	4 J	9.9	6.8	4 J	1 U	1 U	5.2	1 U	1 U	1 U	8.2	9.9
Chromium, total	100[a]	0.47 J	0.69 J	0.54	0.48 J	0.47 J	0.52 J	0.69 J	1 U	1 U	1 U	1 U	1 U
Cobalt	1.1[b]	1.9	17	6.7	14	1.9	2.4	17	2.8	1.9	2.1	13	5.4
Iron	2600[b]	48	78000	17521	3700	38 U	10 UJ	8300	48	280	320	78000	32000
Manganese	88[b]	780	13000	4533	13000	2400	1000	10000	2400	780	820	4200	6200

Notes:
Shading - Exceeds initial screening value
J - Estimated value
N - Normal sample
FD - Field Duplicate
NA - Not available
U - Not detected
ug/l - micrograms per liter
[a] USEPA MCL = USEPA Maximum Contaminant Levels (MCLs). USEPA Drinking Water Standards and Health Advisories. May 2009.
[b] USEPA Tapwater RSL = USEPA Regional Screening Levels for Tapwater. May 2010. Values adjusted for a target hazard quotient of 0.1 to account for cumulative effects on the same target organ.

SAP Worksheet #11: Project Quality Objectives/Systematic Planning Process Statements

(UFP-QAPP Manual Section 2.6.1)

The planned investigation at CCRF (Site 4) is intended to collect additional data to assess the presence of metals in groundwater to complete the SASE. The project quality objectives (PQOs) were developed for this investigation based on the *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA QA/G-4) (USEPA, 2006) and are presented below.

Step 1 – Problem Statement

A Preliminary Assessment (PA) conducted at CCRF (Tetra Tech, 2005) concluded that constituents were present at CCRF that may pose potential risk/hazard above target levels to human health and the environment. Please refer to Section 10.4 of the parent SAP included as Appendix A for further detail regarding analytical results from the PA. Based on the analytical results from the PA, an SASE was implemented at the site, which included additional site investigation. Based upon the preliminary results of the SASE, it was determined that metals in groundwater (As, Cr, Co, Fe, Mn) may pose an unacceptable risk. Therefore, additional data are necessary to confirm the presence of these metals in groundwater, and if present, to evaluate whether their presence is site-related or influenced by natural geochemical and biological conditions.

Step 2 – Study Goals

The objective of the additional investigation is to collect the additional groundwater data necessary to fill data gaps and complete the SASE. Project Action Limits (PALs) presented in Worksheet #15 were developed to provide analytical results that are sufficient for risk screening and/or risk assessment if that is desired. The PALs are not intended as screening levels or cleanup levels; they are used exclusively to select detection limits that allow the analytical results to be used for multiple purposes if needed. The study goals for this SAP Addendum are focused on assessing whether metals in groundwater are site-related or are influenced by natural geochemical and biological conditions. A Tech Memo will be prepared to present the additional groundwater data and associated evaluation. The Tech Memo will then be used as appendix to finalize the SASE.

Step 3 – Information Inputs

Information inputs will consist of environmental screening data and field samples from CCRF, as presented in this SAP Addendum. Figure 2 depicts the existing network of monitoring wells, as well as the locations of the two (2) proposed monitoring wells to be sampled as part of this investigation. The following information inputs will be compiled to characterize the nature of metals in groundwater at the site:

- Field parameters – pH, specific conductivity, turbidity, temperature, dissolved oxygen (DO), ferrous iron, and oxidation-reduction potential (ORP) in groundwater.
- Groundwater sampling – Metals (As, Cr, Co, Fe, Mn, Al, Ca, Mg, K, Na), hexavalent chromium (Cr^{+6}), alkalinity (bicarbonate and carbonate), chloride, nitrate/nitrite, sulfate, TOC, total dissolved solids (TDS), and ammonia.

The specific geochemical and biological indicator measurements and parameters, as well as the oxidation states of Fe and Cr, listed above will be used to aid in evaluating whether the five metals showing potential risk in groundwater (As, Cr, Co, Fe and Mn) are present due to naturally occurring conditions or site-related activities.

Step 4 – Study Boundaries

The CCRF consists of approximately 8 acres of vacant land. Refer to Figure 1 for the regional location and Figure 2 for pertinent site features. Spatial and temporal boundaries are described in the parent SAP, Section 11.3 (Appendix A). There are no deviations or refinements to the boundaries defined in the parent SAP.

Step 5 – Analytical Approach

The analytic approach for the planned investigation is comprised of a series of "if… then…" statements.

- If new monitoring wells are located hydraulically upgradient of the rubble fill area, at locations outside of the projected rubble fill area based on historic aerial photographs and soil sample observations from borings and test pits, where subsurface rubble is absent or less than two (2) feet thick, and where historic wetland environments may have been present, then analytical results collected from the proposed wells will be considered representative of background/upgradient conditions. As shown on Figures 2 and 4, the locations of the proposed wells are based on these criteria.
- If the concentrations of the five metals of primary interest (As, Cr, Co, Fe and Mn) in site groundwater are shown to be consistent with upgradient groundwater, then these metals may be considered to be consistent with local background conditions.
- If the concentrations of metals in groundwater are present in their reduced oxidation states, and are highest in areas of increased organic content based on visual soil descriptions from borings and analytical results of TOC and/or TPH is soil and groundwater results (including natural organic layers as well as the presence of petroleum related compounds), decreased pH, and/or reductive (lower oxygen) conditions, comparative nitrate and nitrite

concentrations, then their solubility and concentration in groundwater may be considered increased by local subsurface conditions.

- If concentrations of hexavalent chromium in site groundwater are found to be below the applicable RSL, and total chromium concentrations are consistent with those collected during the SASE, then the chromium concentrations detected in groundwater in the SASE will be considered to contain hexavalent chromium at concentrations below the RSL.

Step 6 – Performance Criteria

The objective of this section is to complete the following:

- Identify potential sources of study error (e.g., field error, analytical error, etc.);
- Establish and identify the methods used to reduce potential sources of error; and
- Determine how decision errors will be managed during the project.

Sampling Strategy

The groundwater sampling design was developed to further characterize contaminant concentrations historically detected in groundwater at the site. The existing monitoring well network will be sampled as part of this investigation. In addition, two additional groundwater monitoring wells will be installed to provide additional groundwater quality data upgradient of the site. This additional data point will serve as a reference location for comparison to on-site data.

Potential Sources of Error

Potential sources of error may be divided into two main categories: sampling errors and measurement errors. A sampling error can occur when the sampling design, planning, and implementation do not provide for a representative range of heterogeneity at the site. A measurement error can occur when performance variance from laboratory instrumentation, analytical methods, and/or operator error occurs. USEPA identifies the combination of these errors as a "total study error" (USEPA, 2006). One objective of the planned investigation is to reduce the total study error so that decision-makers can be confident that the data collected appropriately represent the chemical characteristics of the site.

Managing Decision Error

The investigation will utilize decision-error minimization techniques in sampling methodologies and laboratory measurement of compounds of interest. Possible decision errors will be minimized during the field investigation by using the following methods:

- Use standard field sampling methodologies (as discussed in Worksheets #18, #20, #21 and #22). Perform sampling activities in accordance with the standard operating procedures (SOPs) referenced in this SAP.

Possible decision errors generated by laboratory measurement errors will be minimized by using the following methods:

- Use applicable analytical methods (discussed in Step 7 below and Worksheets #23, #24, and #25) for sample analysis by a competent analytical laboratory certified by the DoD Environmental Laboratory Accreditation Program (ELAP) to reduce measurement errors.
- Confirm analytical data to identify and control potential laboratory error and sampling error by using spikes, blanks, and replicated samples.

Step 7 – Obtaining the Data

The sampling design for CCRF was developed to optimize resources and generate data to satisfy the PQOs. The critical objective is to obtain a quality dataset for evaluation in the Tech Memo and finalization of the SASE.

Field Parameters

Field measurements will be recorded in a field logbook and/or onto field data collection sheets. Groundwater will be measured in the field for water level, pH, specific conductivity, turbidity, temperature, DO, ferrous iron, and ORP. Field data will be compiled and stored in project folders, for subsequent use in evaluating analytical data and completing the Tech Memo (and ultimately finalizing the SASE).

Groundwater Sampling

Groundwater samples will be collected from the existing monitoring well network, as well as two additional monitoring well locations to be installed as part of this investigation. Please refer to Figure 2 for the locations of the existing monitoring wells, as well as the proposed monitoring wells. The new monitoring wells will be installed hydraulically upgradient of the site and will be screened across the sand and gravel unit below the fill. Depth to water is expected to range from approximately 4 to 13 feet below surface grade (BSG).

In total, ten groundwater monitoring wells will be sampled for select metals (As, Cr, Co, Fe, Mn, Al, Ca, Mg, K, Na), Cr^{+6}, alkalinity (bicarbonate and carbonate), chloride, nitrate/nitrite, sulfate, TOC, TDS, and ammonia. Further details on the groundwater sampling are provided in Worksheet #14 and #17.

Analytical Laboratory Sample Management

The sample matrix, number of samples, and number and type of laboratory quality assurance and quality control (QA/QC) samples are summarized in the "Sample Details Table" of this SAP. Details on the analytical group, sample volumes, sample container specifications, preservation requirements, and maximum holding times are identified in Worksheet #19 and #20 of this SAP.

The laboratory will provide EQuIS electronic data deliverable files, portable document format (PDF) files of the data deliverables for all project data, and a hard copy of data deliverables for all results including results from secondary subcontract laboratories. Designated samples will be used to obtain necessary subsamples for laboratory QC measurements, which includes analytical sample duplicate and sample matrix spike/matrix spike duplicate (MS/MSD). Tasks will be completed using the laboratory SOPs.

Resolution will provide data validation services and verify and evaluate the usability of the data as identified in Worksheets #31 through #37.

PDF copies of all analytical data packages will be stored on CD-ROM, archived in the NAVFAC LANT Administrative Record, and uploaded onto the Naval Installation Restoration Information Solution (NIRIS) system using the Navy Electronic Data Deliverable (NEDD) database format. All other data generated in the field and reports generated for the project will be stored as computer readable data files by Resolution.

SAP Worksheet #12-1 Measurement Performance Criteria – Field QC Samples

(UFP-QAPP Manual Section 2.6.2)

Matrix: Groundwater

Analytical Group: ICP-MS Metals

Concentration Level: Low

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Equipment Rinsate Blank	ICP-MS Metals	One per day of sampling per type of equipment used	Accuracy/Bias	No target metals > ½ LOQ (> LOQ for common laboratory contaminants) and > 1/10 the amount measured in any sample or 1/10 the PAL, whichever is greater. For negative blanks, absolute value < LOD. Blank result must not otherwise affect sample results (see DoD QSM Box D-1).
Field Duplicate		One per ten samples per matrix	Precision	RPD ≤ 30% if both results are ≥5 x LOQ. |Diff| ≤ LOQ if values are < 5x LOQ
Matrix Spike		Submitted: One per twenty samples per matrix. Analyzed: Most frequent of one per twenty samples or SDG per matrix, or one per prep batch per matrix.	Accuracy/Bias	Refer to Worksheet #28-1

SAP Worksheet #12-2 Measurement Performance Criteria – Field QC Samples

(UFP-QAPP Manual Section 2.6.2)

Matrix: Groundwater

Analytical Group: ICP-AES Metals

Concentration Level: Low

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Equipment Rinsate Blank	ICP-MS Metals	One per day of sampling per type of equipment used	Accuracy/Bias	No target metals> ½ LOQ (> LOQ for common laboratory contaminants) and > 1/10 the amount measured in any sample or 1/10 the PAL, whichever is greater. For negative blanks, absolute value must be < LOD. Blank result must not otherwise affect sample results (see DoD QSM Box D-1).
Field Duplicate		One per ten samples per matrix	Precision	RPD ≤ 30% if both results are ≥5 x LOQ. |Diff| ≤ LOQ if values are < 5x LOQ
Matrix Spike		Submitted: One per twenty samples per matrix. Analyzed: Most frequent of one per twenty samples or SDG per matrix, or one per prep batch per matrix.	Accuracy/Bias	Refer to Worksheet #28-2

SAP Worksheet #12-3 Measurement Performance Criteria – Field QC Samples

(UFP-QAPP Manual Section 2.6.2)

Matrix: Groundwater

Analytical Group: Hexavalent Chromium

Concentration Level: Low

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Equipment Rinsate Blank	Hexavalent Chromium	One per day of sampling per type of equipment used	Accuracy/Bias	No hexavalent chromium > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the PAL, whichever is greater. Blank result must not otherwise affect sample results (see DoD QSM Box D-1).
Cooler Temperature Indicator		One per cooler	Accuracy/Representativeness	Temperature ≤ 6 degrees Celsius.
Field Duplicate		One per ten samples per matrix	Precision	RPD ≤ 30% if both results are ≥5 x LOQ; If < 5 x LOQ, |Diff|≤ LOQ
Matrix Spike		Submitted: One per twenty samples per matrix. Analyzed: Most frequent of one per twenty samples or SDG per matrix, or one per prep batch per matrix.	Accuracy/Bias	Refer to Worksheet #28-3

SAP Worksheet #12-4 Measurement Performance Criteria – Field QC Samples

(UFP-QAPP Manual Section 2.6.2)

Matrix: Groundwater

Analytical Group: Carbonate and Bicarbonate Alkalinity

Concentration Level: Low

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Cooler Temperature Indicator	Alkalinity	One per cooler	Accuracy/Representativeness	Temperature ≤ 6 degrees Celsius.
Field Duplicate		One per ten samples per matrix	Precision	RPD ≤ 30 if both results are >3 x LOQ; RPD ≤100% for samples ≤ 3 x LOQ
Matrix Spike		Submitted: One per twenty samples per matrix. Analyzed: One per ten samples.	Accuracy/Bias	Refer to Worksheet #28-4

SAP Worksheet #12-5 Measurement Performance Criteria – Field QC Samples

(UFP-QAPP Manual Section 2.6.2)

Matrix: Groundwater

Analytical Group: Anions (Chloride, Nitrate, Nitrite, Sulfate)

Concentration Level: Low

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Cooler Temperature Indicator	Anions (Chloride, Nitrate, Nitrite, Sulfate)	One per cooler	Accuracy/Representativeness	Temperature ≤ 6 degrees Celsius.
Field Duplicate		One per ten samples per matrix	Precision	RPD ≤ 30 if both results are >3 x LOQ; RPD ≤100% for samples ≤ 3 x LOQ
Matrix Spike/Matrix Spike Duplicate		Submitted and Analyzed: One per twenty samples per matrix.	Accuracy/Bias/Precision	Refer to Worksheet #28-5

SAP Worksheet #12-6 Measurement Performance Criteria – Field QC Samples

(UFP-QAPP Manual Section 2.6.2)

Matrix: Groundwater

Analytical Group: Ammonia

Concentration Level: Low

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Cooler Temperature Indicator	Ammonia	One per cooler	Accuracy/Representativeness	Temperature ≤ 6 degrees Celsius.
Field Duplicate		One per ten samples per matrix	Precision	RPD ≤ 30 if both results are >3 x LOQ; RPD ≤100% for samples ≤ 3 x LOQ
Matrix Spike/Matrix Spike Duplicate		Submitted and Analyzed: One per twenty samples per matrix.	Accuracy/Bias/Precision	Refer to Worksheet #28-6

`

SAP Worksheet #12-7 Measurement Performance Criteria – Field QC Samples

(UFP-QAPP Manual Section 2.6.2)

Matrix: Groundwater

Analytical Group: Total Organic Carbon (Method 9060)

Concentration Level: Low

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Cooler Temperature Indicator	Total Organic Carbon (Method 9060)	One per cooler	Accuracy/Representativeness	Temperature ≤ 6 degrees Celsius.
Field Duplicate		One per ten samples per matrix	Precision	RPD ≤ 30 if both results are >3 x LOQ; RPD ≤100% for samples ≤ 3 x LOQ
Matrix Spike		Submitted: One per twenty samples per matrix. Analyzed: One per ten samples per matrix.	Accuracy/Bias	Refer to Worksheet #28-7

SAP Worksheet #12-8 Measurement Performance Criteria – Field QC Samples

(UFP-QAPP Manual Section 2.6.2)

Matrix: Groundwater

Analytical Group: Total Dissolved Solids

Concentration Level: Low

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Cooler Temperature Indicator	Total Dissolved Solids	One per cooler	Accuracy/Representativeness	Temperature ≤ 6 degrees Celsius.
Field Duplicate		One per ten samples per matrix	Precision	RPD ≤ 30 if both results are >3 x LOQ; RPD ≤100% for samples ≤ 3 x LOQ

SAP Worksheet #12: Notes

(UFP-QAPP Manual Section 2.6.2)

|Diff| – Absolute Difference
DoD QSM – Department of Defense Quality Systems Manual
DQI – Data Quality Indicator
LOD – Limit of Detection
LOQ – Limit of Quantitation
PAL – Project Action Limit
RPD – Relative Percent Difference
SDG – Sample Delivery Group

SAP Worksheet #13: Secondary Data Criteria and Limitations Table

(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Soil, Groundwater and Sediment Data	Tetra Tech NUS, Inc., Revised Draft Study Area Screening Investigation Report, May 2012.	Tetra Tech NUS, Inc., Groundwater Data, November - December 2010.	Data will be reviewed and incorporated, as necessary, into the conclusions of the Tech Memo for Coddington Cove.	Historical reporting limits may not be sufficiently low to meet project action limits for this investigation.

SAP Worksheet #14: Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

Site Visit

Prior to the initiation of intrusive field work, Resolution, the Navy and the regulatory agencies will conduct a site visit to mark out the locations of the proposed monitoring wells. The mark out of the locations will be utilized for utility clearance (described below).

Utility Clearance

Prior to the initiation of intrusive field work, utility clearance will be conducted in accordance with SOP 3-01.

Drilling and Monitoring Well Installation

Two monitoring wells will be installed along the upgradient boundary of the site as shown of Figure 2. The locations are projected to be outside or on the edge of the rubble fill area. If anthropomorphic material is encountered in the upper ten feet, the boring will be abandoned and a second boring will be attempted at an adjacent location approximately five feet to the west or east along the fence line. If fill is encountered at this second location, an additional boring will be advanced. The process will be repeated as need up to a maximum of six borings at each proposed well locations. If less than two feet of fill is encountered at the final boring, the monitoring well will be installed. The soil borings will be advanced utilizing hollow stem auger drilling methods and will be completed as a groundwater monitoring wells. The monitoring wells will provide upgradient reference locations so that groundwater quality can be compared to the groundwater within the limits of the rubble fill area. Please refer to Figure 2 for the locations of the proposed monitoring wells.

During advancement of the soil borings, soil samples will be continuously collected via split spoons or hand auger for visual description of soil composition in accordance with SOP 3-16 and for headspace screening utilizing a PID in accordance with SOP 3-19. The field screening will not be utilized to determine well construction or to screen for potential contaminants of concern but rather to ensure the monitoring well will be placed in a sufficient location to be used as an upgradient, reference point. Soil samples will not be collected for laboratory analysis as part of this field investigation.

The soil borings will be advanced to a depth of approximately 20 feet. The final depth will be determined in the field. The soil borings will be completed as monitoring wells. The monitoring wells will be constructed with a 10-foot screen installed across sand and gravel unit to be consistent with the existing on-site monitoring well network (Table 17-1). The monitoring well filter pack will

be designed based on the formation encountered and the well screen slot size selected. The monitoring wells will be constructed in accordance with SOP 3-12 and RIDEM regulations (RIDEM, 2010 and RIDEM, 2011). Soil cuttings will be containerized as IDW and properly disposed of.

Monitoring Well Development

Following installation, the newly installed monitoring wells, as well as the existing monitoring wells, will be developed/re-developed in accordance with SOP 3-13. Per RIDEM regulations, well development will not commence until at least 24 hours after well installation. The existing monitoring wells will be re-developed to reduce the turbidity to the lowest condition possible for the formation. In compliance with RIDEM regulations, the monitoring wells will be developed until turbidity is equal to or less than 5 nephlometric turbidity units (NTU), if possible. If turbidity less than 5 NTUs is not possible, development will continue until turbidity readings are stability in accordance with SOP 3-13. Development fluids will be containerized as Investigative-Derived Waste (IDW) and properly disposed of.

Groundwater Quality Monitoring and Sample Collection

Following development, the monitoring wells will be allowed to stabilize for at least 1 week prior to groundwater gauging and sampling. The monitoring wells will be gauged and sampled in accordance with SOP 3-14 and USEPA Region 1 Low-Flow Sampling Guidance (USEPA, 1996).

Groundwater will be measured in the field for water level, pH, specific conductivity, turbidity, temperature, DO, ferrous iron, and ORP, and will be analyzed in the field for ferrous iron. Groundwater samples will also be transported off-site for analysis of select metals (As, Cr, Co, Fe, Mn, Al, Ca, Mg, K, Na) via USEPA Methods 6010 and/or 6020, hexavalent chromium, TOC, TDS, alkalinity, chloride, nitrate/nitrite, sulfate, and ammonia. Purge water will be containerized as IDW and properly disposed of.

Quality Assurance/Quality Control

The QA/QC sample collection frequency is as follows (also shown in Worksheet #20):

- **Equipment blanks** - 1 per day per type of sampling equipment used for those methods and matrices for which they are included. Equipment blanks will not be submitted for the analyses presented for geochemical and biological indicator characterization of groundwater.
- **Field duplicates** – (single blind samples) 1 per 10 samples per method and matrix
- **MS/MSD or MS/Matrix Duplicate** - 1 per 20 samples per method and matrix

Equipment Decontamination

To the maximum extent possible, Resolution will utilize dedicated and disposable sampling equipment to avoid the potential for cross contamination of samples due to inadequate decontamination processes. The dedicated/disposable sampling equipment will include a disposable Teflon® and polyethylene tubing, disposable gloves, and laboratory supplied sample bottles.

Non-disposable or non-dedicated sampling equipment (e.g., auger flights, split spoon samplers, surge blocks, peristaltic pumps, water level indicators, water quality meters, etc.) will be decontaminated prior to sampling and between samples. Cleaning of equipment is performed to prevent cross-contamination between samples and to maintain a clean working environment for all personnel. Decontamination will generally consist of a water rinse station to remove gross contamination (if needed), followed by a non-phosphate detergent (e.g., Alconox®) water rinse, and a rinse with de-ionized water. If smaller equipment is to be stored or transported, it will be wrapped in aluminum foil after air-drying. The auger flights associated with the hollow stem auger will be decontaminated after the bore hole and the split spoon samplers will be decontaminated after each sample collection. All decontamination water generated during decontamination of sampling equipment will be containerized as IDW and properly disposed of.

Investigation-Derived Waste Management

The IDW, consisting of soil cuttings, purge/well development water, water generated during decontamination processes and PPE, generated during well development/sampling and decontamination activities will be collected in properly labeled 55-gallon drums, sampled for waste characterization, and temporarily staged on-site for subsequent off-site disposal.

Laboratory Coordination, Data Management and Validation

Resolution's Project Chemist will track the samples from collection through analysis and obtaining data packets from the laboratories within the appropriate turnaround time (TAT) of sample receipt. A signed certificate of analysis will be provided in the narrative section of each laboratory data package. The laboratory will submit the data in hard copy and an electronic format per the Electronic Data Deliverable (EDD) format specified by Resolution.

Analytical results will be validated according to the procedures in Worksheet #36. The validated analytical data will then be converted and/or verified as compliant with the Navy's NEDD format, and uploaded into the Navy's NIRIS database. Other data generated in the field, as well as reports

produced for this project, will be uploaded into appropriate portions of NIRIS, packaged in the appropriate formats (e.g., hard copy and/or electronic), and submitted to the appropriate document distribution lists provided by the Navy.

Report Preparation

Following data collection, analysis and validation, a Tech Memo will be prepared for the CCRF Area. The Tech Memo elements will include a summary of field efforts, deviations from the SAP (if any), data tables and figures and comprehensive discussion and interpretation of the groundwater sampling results/geochemical analyses.

SAP Worksheet #15: Establishing Laboratory Reference Limits

(UFP-QAPP Manual Section 2.8.1)

The following table identifies the PALs and comparison of the PALs to analytical laboratory reference limits (i.e., LODs, LOQs and DLs). The PALs represent the limits of detection that analytical data must meet in order to be of sufficient quality for use in the Tech Memo/SASE. These levels are designed to represent the lowest of the relevant human health and ecological screening levels and other applicable criteria that will be used in the SASE. The PALs are not intended to be used as cleanup levels or to identify the presence of risk/hazard. The sources considered for development of PALs are referenced at the bottom of the following table.

Table 15-1
Project Action Limits (PALs) - Groundwater
CCRF Area
NAVSTA, Newport, RI

Analytical Method	Compound	CAS	Project Action Limits for Groundwater (ug/L) (a)		Laboratory Reference Limits (ug/L)					
			Value	Source (b)	LOQs	Is LOQ >PAL (Y/N)?	LODs	Is LOD>PAL (Y/N)?	DLs	Is DL>PAL (Y/N)?
TAL Metals via SW6010	Aluminum	7429-90-5	1,600	USEPA Tapwater RSL	300	N	100	N	14.8	N
TAL Metals via SW6020	Arsenic	7440-38-2	0.045	USEPA Tapwater RSL	5	Y	4	Y	2.2	Y
TAL Metals via SW6010	Calcium	7440-70-2	NA		100	N	80	N	20	N
TAL Metals via SW6020	Chromium	7440-47-3	0.031	USEPA Tapwater RSL	5	Y	4	Y	0.22	Y
TAL Metals via SW6020	Cobalt	7440-48-4	0.47	USEPA Tapwater RSL	1	Y	0.3	N	0.06	N
TAL Metals via SW6010	Iron	7439-89-6	1,100	USEPA Tapwater RSL	100	N	60	N	13	N
TAL Metals via SW6010	Magnesium	7439-95-4	NA		100	N	80	N	7.8	N
TAL Metals via SW6020	Manganese	7439-96-5	32	USEPA Tapwater RSL	2	N	1	N	0.35	N
TAL Metals via SW6010	Potassium	7440-09-7	NA		1000	N	400	N	31	N
TAL Metals via SW6010	Sodium	7440-23-5	NA		1000	N	400	N	18	N
SW-846 7196A	Hexavalent Chromium	18540-29-9	0.031	USEPA Tapwater RSL	25	Y	12.5	Y	0.76	Y
SW-846 9056A	Nitrate-N	14797-55-8	2,500	USEPA Tapwater RSL	50	N	25	N	12	N
SW-846 9056A	Nitrite-N	14797-65-0	160	USEPA Tapwater RSL	50	N	25	N	10.2	N
SW-846 9056A	Chloride	16887-00-6	250,000	USEPA MCL	2000	N	1000	N	100	N
SW-846 9056A	Sulfate	14808-79-8	250,000	USEPA MCL	1000	N	500	N	90	N
EPA 350.1	Ammonia-N	NA	NA		100	N	50	N	11	N
SM2540C	Total Dissolved Solids (TDS)	NA	NA		10000	N	5000	N	5000	N
SM5310B	Total Organic Carbon (TOC)	NA	NA		1000	N	500	N	102	N
SM2320B	Carbonate Alkalinity	3812-32-6	NA		5000	N	2500	N	228	N
SM2320B	Bicarbonate Alkalinity	71-52-3	NA		5000	N	2500	N	228	N

Notes:
CCRF - Coddington Cove Rubble Fill
CAS - Chemical Abstracts Service
DL - Detection Limit
LOD - Limit of Detection
LOQ - Limit of Quantitation
NA - Not available/applicable
PAL - Project Action Limit
ug/L - microgram per liter
TAL - Target Analyte List

Highlighting indicates the compound's laboratory reference value(s) is/are above the selected PAL.
(a) The PAL represents the limits of detection that analytical data must meet in order to be of sufficient quality for use in the Technical Memorandum.
(b) Groundwater PALs were selected based on the lower of the following sources of screening levels:
RIDEM M1 GA = RIDEM, 2011. Method 1 GA Groundwater Objectives. Presumable to be suitable for drinking water use without treatment.
RIDEM M1 GB = RIDEM, 2011. Method 1 GB Groundwater Objectives. Presumed not suitable for use as a current or potential source of drinking water.
USEPA Tapwater RSL = USEPA Regional Screening Levels for Tapwater. May 2013. Values adjusted for a target hazard quotient of 0.1 to account for cumulative effects on the same target organ.
USEPA VI = USEPA Target Groundwater Concentrations for the Vapor Intrusion Pathway. Calculated using the USEPA Vapor Intrusion Screening Level Calculator. Version 3.1, June 2013 RSLs.
USEPA MCL = USEPA Maximum Contaminant Levels (MCLs). USEPA Drinking Water Standards and Health Advisories. EPA 822-R-06-013. April 2012.

SAP Worksheet #16: Project Schedule/Timeline Table

(UFP-QAPP Manual Section 2.8.2)

Deliverable	Timeframe	Planned Date
Field Program, Data Evaluation		
Complete Field Program	30 days after Final SAP Addendum	12/31/2013
Complete Laboratory Analysis	21-day standard TAT	1/30/2014
Complete Data Validation and Import	45 days for data validation	3/1/2014
Complete Internal Draft Tech Memo	24 days for report preparation	3/31/2014
Technical Memorandum		
Internal Draft to Navy	Upon completion of Field Program and Data Evaluation	3/31/2014
Receive Navy Comments	21 days after Internal Draft	4/21/2014
Draft to Agencies	14 days after receipt of comments	5/5/2014
Receive Agency Comments	30 days after Draft	6/4/2014
Navy RTC to Agencies	30 days after receipt of comments	7/4/2014
Agency Concurrence	30 days after RTC to Agencies	8/3/2014
Final to Tetra Tech as Appendix to SASE	60 days after RTC to Agencies	9/2/2014

SAP Worksheet #17: Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

Overview

This section describes the sampling rationale and sampling design for the collection of groundwater samples at CCRF. Groundwater samples will be collected from the existing monitoring well network, as well as two newly installed monitoring wells. The data will ultimately be used to prepare a Tech Memo and to support the SASE to be finalized for this site. The planned investigation is designed to collect additional groundwater data to better define the nature of site-related impacts. Analytical parameters to achieve the specific goals listed above are summarized in the following table:

Table 17-1
Analytical Parameters Designed to Achieve Investigation Goals
CCRF, NAVSTA Newport, RI

Analytical Parameter	Matrix
Parameters of Interest	
Metals (As, Cr, Co, Fe, Mn)	GW
Hexavalent chromium (Cr^{+6})	GW
Geochemical and Biological Indicator Data	
TOC	GW
TDS	GW
Alkalinity (bicarbonate and carbonate)	GW
Chloride, nitrite/nitrate, sulfate	GW
Ammonia	GW
Metals (Al, Ca, Mg, K, Na)	GW

Notes:
GW – Groundwater
TAL – Target Analyte List

Further details regarding the specific DQOs for the planned investigation are presented in Worksheet #11, and PALs are quantified in Worksheet #15. The PALs represent the limits of detection that analytical data must meet in order to be of sufficient quality for use in the Tech Memo and the SASE. These levels are designed to be equal to or lower than relevant human health and ecological screening levels. The PALs are not intended to be used as cleanup levels or

to identify the presence of risk/hazard. The following paragraphs describe the general methodology and rationale for the specific sampling approach.

Field Parameters

Field parameters will consist of measurements associated with this sampling program. Measurements of pH, specific conductivity, turbidity, temperature, DO, ferrous iron, and ORP will be recorded in relevant field logs. The specific methodology and procedures to be implemented for field parameters are included within the SOPs provided with this SAP.

Groundwater Sampling

Two groundwater monitoring wells will be constructed during the planned investigation at CCRF. The monitoring wells will be installed to provide hydraulically upgradient reference locations so that groundwater quality can be compared to the groundwater quality on-site. Proposed monitoring well locations (MW-09 and MW-10) are present on Figure 2. The criteria used to select suitable monitoring well locations included:

- Hydraulically upgradient of the current monitoring well network
- Outside of the rubble fill areas
- Where historic wetlands may have been present
- On site to eliminate the uncertainty associated with off-site sources, such as access issues and the potential presence of unrelated impacts.

Historic aerial photographs from 1939, 1951-1952 (Tetra Tech), 1962, 1965 (Tetra Tech), and 1981 were reviewed to determine predevelopment surface conditions and to estimate the extent of rubble filling performed across the site. The aerial photos are included in Appendix D. The 1939 aerial photo indicates that wetland/marshes were historically located along the northwestern part of the site, and branches of the marsh may have been present across other parts of the site. The topographic map of the present day ground surface (generated with TOPO! from National Geographic) shows that the area persists as a regional low and drainage area. The aerial photos from 1962, 1965, and 1981 indicate that rubble fill areas were located in the central and western portions of the site, and did not extend to Coddington Highway. Cross-section B-B' on Figure 4 presents a consistent interpretation which shows the fill unit pinching out as it approaches the highway to the south along the southern border of the site. Based on this evaluation, the proposed well locations meet the criteria listed above.

During well installation, soil samples for description and screening will be collected continuously to assess the presence of anthropomorphic fill material that is characteristic of the CCRF fill (brick, concrete, asphalt, metal, etc.). If these materials are absent, then the location will be considered

to be located outside of the rubble fill area. If these material are present but in an interval less than two (2) feet thick, then the location will be considered acceptable to assess subsurface upgradient groundwater conditions. If more than two (2) feet of these materials are encountered, the boring will be abandoned and a second boring will be attempted at a location five feet to the west or east. If fill is encountered at this second location, an additional boring will be advanced. The process will be repeated as need up to a maximum of six borings and each proposed well locations. Each well will be constructed with a 10-foot well screen installed in the sand and gravel unit, consistent with the majority of the existing monitoring wells. Construction details for the existing monitoring well network and the proposed monitoring wells are presented in Table 17-1.

In total, groundwater samples will be collected from ten monitoring wells. The existing monitoring wells will be re-developed to reduce the turbidity to the lowest condition possible for the formation. In addition, the newly installed monitoring wells will be developed after installation; at least 24 hours will be allowed between installation and development. After development, monitoring wells will be allowed to stabilize for at least 1 week, before returning to the monitoring wells for groundwater sampling. The specific methodology and procedures to be implemented for monitoring well development and sampling are included within the SOPs provided with this SAP.

The intent of the groundwater sampling program is to assess the presence/concentrations of metals in groundwater underlying CCRF as well as hydraulically upgradient of the site. In addition, key geochemical and biological indicator parameters will be collected to assess/aid in the determination of the source and potential influences on the metals in groundwater.

The existing site monitoring wells (8) will be sampled as well as the two (2) newly installed upgradient monitoring wells. Groundwater samples will be collected and handled in accordance with SOP 3-24 and USEPA Region 1 Low-Flow Sampling Guidance and analyzed for select metals (As, Cr, Co, Fe, Mn, Al, Ca, Mg, K, Na), hexavalent chromium (Cr^{+6}), chloride, sulfate, nitrate/nitrite, alkalinity (bicarbonate and carbonate), TOC, TDS and ammonia. In addition to the primary samples, QA/QC samples will be collected in accordance with this SAP.

Samples will be shipped on the day of sampling to accommodate hold times as documented in Worksheet #19. The samples will be preserved, chilled, etc. in accordance with this SAP. All sample shipments to a designated laboratory will contain appropriate COC forms.

If warranted, subsequent rounds of groundwater sampling could be implemented using the same procedures outlined in this SAP.

TABLE 17-1
Monitoring Well Construction Details
Coddington Cove Rubble Fill Area (IR Site 4)
NAVSTA Newport, Newport RI

						REFERENCE ELEVATIONS (msl)			WATER LEVEL 12/20/2010		
WELL	DIAM. (inches)	Well Type	SCREENED INTERVAL (feet bgs)	SCREEN ELEVATION (NGVD 29)	Top of PVC (feet ags)	GROUND (NGVD 29)	CASING (NGVD 29)	INNER PVC (NGVD 29)	WATER DEPTH (GROUND)	WATER DEPTH (TOP of PVC)	ELEVATION (NGVD 29)
MW01	2	WT	5 to 15	13.4 to 3.4	2.91	18.4	21.53	21.31	8.19	11.10	10.21
MW02	2	WT	4 to 14	14.5 to 4.5	3.43	18.5	22.22	21.93	7.91	11.34	10.59
MW03	2	WT	4 to 14	14.1 to 4.1	2.88	18.1	21.28	20.98	8.38	11.26	9.72
MW04	2	WT	5 to 15	13.3 to 3.3	2.81	18.3	21.38	21.11	9.24	12.05	9.06
MW05	2	Below WT	9 to 19	8.4 to -2.6	2.90	17.4	20.45	20.30	7.04	9.94	10.36
MW06	2	Below WT	4 to 14	7.7 to -2.3	3.04	11.7	15.00	14.74	1.73	4.77	9.97
MW07	2	WT	5 to 15	11.4 to 1.4	3.04	16.4	19.67	19.44	9.26	12.30	7.14
MW08	2	WT	6 to 16	11.0 to 1.0	2.55	17.0	19.86	19.55	7.9	10.45	9.10
MW09	2	TBD	~9 to 19	TBD	TBD	TBD	TBD	TBD	TBD	TBD	TBD
MW10	2	TBD	~9 to 19	TBD	TBD	TBD	TBD	TBD	TBD	TBD	TBD

NOTES: Horizontal Datum - NAD 1983
Vertical Datum - NGVD 1929 (msl)
WT - Well screened across the water table.
Below WT - Well screened below the water table.
bgs - below ground surface
ags - abve ground surface
TBD - To Be Determined

SAP Worksheet #18: Location-Specific Sampling Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples	Sampling SOP Reference
MW01 through MW10	Groundwater	Base of fill and/or within the sand and gravel unit	Metals (As, Cr, Co, Fe, Mn, Al, Ca, Mg, K, Na), hexavalent chromium, TOC, TDS, alkalinity (bicarbonate and carbonate), chloride, nitrate/nitrite, sulfate, ammonia	10	3-14 Monitoring Well Sampling

SAP Worksheet #19: Field Sampling Requirements Table[1]

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group[2]	Analytical and Preparation Method / SOP Reference[3]	Containers (number, size, and type)	Sample Volume (units)[4]	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[5] (preparation / analysis)
Groundwater	ICP-AES Metals	SW846 3010A, 6010C / CA-604, CA-608	One 250-ml wide-mouth HDPE bottle	50 ml	HNO_3 to pH<2	6 months to analysis
	ICP-MS Metals	SW846 3010A, 6020A / CA-604, CA-627	One 250-ml wide-mouth HDPE bottle	50 ml	HNO_3 to pH<2	6 months to analysis
	Hexavalent Chromium	SW7196 / KAS SOP CA-746	One 250-ml wide-mouth HDPE bottle	125 ml	Cool to ≤ 6 °C; no headspace	24 hours to analysis
	Carbonate and Bicarbonate Alkalinity	SM 2320 B / CA-739	One 100-ml wide-mouth HDPE bottle	50 ml	Settled; Cool to ≤ 6 °C	14 days to analysis
	Anions (Chloride, Nitrate, Nitrite, Sulfate)	USEPA 300.0 / CA-742	One 100-ml wide-mouth HDPE bottle	1 ml	Cool to ≤ 6 °C	48 hours (nitrate and nitrite) / 28 days (sulfate and chloride) to analysis
	Ammonia (as N) non-distilled	EPA 350.1/CA-770	One 250-ml polyethylene bottle	5mL	H_2SO_4 to pH < 2, Cool to ≤ 6 °C	28 days to analysis
	Total Organic Carbon (Method 9060)	SM 9060 / CA-763	Two 40-ml VOA vials	40 ml	Cool to ≤ 6 °C	28 days to analysis
	Total Dissolved Solids	SM 2540C / CA-719	One 250-ml wide-mouth HDPE bottle	100 ml	Cool to ≤ 6 °C	7 days to analysis

Notes:
1. Only analyses that will be submitted to a fixed laboratory are included. Field analyses for water quality parameters (DO, ORP, temperature, specific conductance), and ferrous iron will be analyzed immediately.
2. Refer to Worksheet #15 for specific target analytes.
3. Refer to the Analytical SOP References table (Worksheet #23).
4. Minimum sample volume requirement.
5. Maximum holding time is calculated from the time the sample is collected to the time the sample is extracted, digested or analyzed.

Abbreviations:
°C – Degrees Celsius
ICP-MS- Inductively Coupled Plasma Mass Spectrometry
ml – milliliter
HDPE – high-density polyethylene
HNO_3 – nitric acid
H_2SO_4 – sulfuric acid
ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy

SAP Worksheet #20: Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Groundwater Investigation

		FIELD SAMPLES				LABORATORY SAMPLES			PER ROUND				TOTALS	
Matrix/Parameter	Analytical Method	Field Samples	Equipment Blank[1]	Trip Blanks	Field Duplicates[2]	MS[3]	MSD[3]	MD[4]	Field Samples[5]	Field Days[7]	SDGs[6]	No. of Rounds	No. of SDGs[6]	No. of Field Samples[5,7]
Groundwater														
ICP-MS Metals (As, Cr, Co, Fe, Mn)	SW846 6020A	10	2	--	1	1	--	1	13	2	1	1	1	13
ICP-AES Metals (Al, Ca, Mg, K, Na)	SW846 6010C	10	2	--	1	1	--	1	13	2	1	1	1	13
Hexavalent Chromium	SW846 7196A	10	2		1	1	--	1	13	2	1	1	1	13

Supplemental Groundwater Characterization

		FIELD SAMPLES				LABORATORY SAMPLES			PER ROUND				TOTALS	
Matrix/Parameter	Analytical Method	Field Samples	Equipment Blank[1]	Trip Blanks	Field Duplicates[2]	MS[3]	MSD[3]	MD[4]	Field Samples[5]	Field Days[7]	SDGs[6]	No. of Rounds	No. of SDGs[6]	No. of Field Samples[5,7]
Groundwater														
Alkalinity	SM2320B	10	--	--	1	1	--	1	11	2	1	1	1	11
Anions (Chloride, Nitrate, Nitrite, Sulfate)	SW846 9056A	10	--	--	1	1	--	1	11	2	1	1	1	11
Total Organic Carbon (Method 9060)	SW846 9060	10	--	--	1	1	--	1	11	2	1	1	1	11
Ammonia	EPA 350.1	10	--		1	1	--	1	11	2	1	1	1	11
Total Dissolved Solids	SM 2540C	10	--	--	1	--	--	1	11	2	1	1	1	11

Notes:

1 - Equipment blanks will be collected at a rate of 1 per day per type of sampling equipment used for those methods and matrices for which they are included in the table above. Equipment blanks will not be submitted for the analyses presented for supplemental characterization for groundwater.
2 - Field duplicates are blind to the laboratory and will be submitted at a rate of 1 per 10 samples per method and matrix for those analyses for which they are included in the table above.
3 - Matrix Spike and Matrix Spike Duplicates will be submitted at a rate of 1 per 20 samples per method and matrix for those analyses for which they are included in the table above.
4 - Matrix Duplicates will be submitted at a rate of 1 per 20 samples per method and matrix for those analyses for which an MS Duplicate is not performed for the methods shown.
5 - Total does not include MS, MSD, or MD.
6 - A SDG consists of no more than 20 samples of the same matrix and method shipped in one sample shipment. It is assumed that samples will be shipped every day.
7 - Sample quantites, QC sample quantities and SDG quantities assume the number of field days shown for each event.

Abbreviations:
ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP-MS- Inductively Coupled Plasma Mass Spectrometry
MS - matrix spike
MD - matrix duplicate
MSD - matrix spike duplicate

SAP Worksheet #21: Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
3-01	Utility Clearance	Resolution	Ground Penetrating Radar, Air Knife	N	
3-02	Log Books	Resolution		N	
3-03	Record Keeping, Sample Labeling, and Chain-of-Custody	Resolution		N	
3-04	Sampling Handling, Storage and Shipping	Resolution		N	
3-05	IDW Management	Resolution		N	
3-06	Equipment Decontamination	Resolution		N	
3-07	Land Surveying	Resolution	GPS	N	
3-12	Monitoring Well Installation	Resolution	HSA Drill Rig	N	
3-13	Monitoring Well Development	Resolution	Surge Block, Bailer, Submersible Pump, Oil Water Interface Probe	N	
3-14	Monitoring Well Sampling	Resolution	YSI, Turbidity Meter, Peristaltic Pump, Oil Water Interface Probe	N	
3-16	Soil and Rock Classification	Resolution		N	
3-18	Field Analysis of Ferrous Iron HACH	Resolution	HACH Ferrous Iron Field Kit	N	
3-19	Headspace Screening for Total VOCs	Resolution	PID	N	
3-20	Operation and Calibration of a PID	Resolution	PID	N	
3-21	Surface and Subsurface Soil Sampling	Resolution	HSA Drill Rig	N	
3-24	Water Quality Parameter Testing	Resolution	YSI, Turbidity Meter, Peristaltic Pump	N	
3-33	Subsurface Soil Sampling by Split Spoon	Resolution	Hollow Stem Auger Drill Rig	N	

SAP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
YSI Water Quality Meter	Calibration	Daily before use and at the end of the day. If readings appear anomalous.	pH: reads ± 0.2 pH of calibration standards (4 and 10 pH). DO: ± 0.3 mg/L of theoretical oxygen solubility (calibrate at 100% water saturated air and 0.0 mg/l standard. Temperature: ± 0.15°C compared to NIST Thermometer. Specific conductivity: ± 5% of standard solution. ORP: ± 10 mv from theoretical standard value at that temperature.	Clean sensors with deionized water and calibrate again. For DO, check barometric pressure setting and site elevation. Do not use instrument if not able to calibrate. properly	Field Team Lead	3-24 Water Quality Parameter Testing	
Turbidity Meter	Calibration	Daily before use and at the end of the day. If readings appear anomalous.	Turbidity: ± 10% of the standards (1.00 and 10.0 NTU)	Wipe tubes and chamber. Test blank three times to ensure consistent readings of 1.00 NTU. Then repeat for 10.0 NTU. Do not use instrument if not able to calibrate properly.	Field Team Lead	3-24 Water Quality Parameter Testing	
Ferrous Iron Field Kit	Calibration	Daily before use and at the end of the day. If readings appear anomalous.	Ferrous Iron: ± 0.1 mg/l of standards solution (0.0 mg/L and 1.00 mg/L)	Clean sample cell and recalibrate.	Field Team Lead	3-18 Field Analysis of Ferrous Iron HACH	
Photoionization Detector	Calibration	Daily before use, every 20 readings, or anytime unstable readings occur.	PID: ±10% of standards (100 ppm Isobutylene, and commercially available zero gas)	Inspect connections to ensure proper seal. Calibrate again. Do not use instrument if not able to calibrate. properly	Field Team Lead	3-20 Operation and Calibration of a PID	

SAP Worksheet #23: Analytical SOP References Table[1]

(UFP-QAPP Manual Section 3.2.1)

Laboratory Name and Address: Katahdin Analytical Services, 600 Technology Way, Scarborough, ME (Please refer to Worksheet #30 for Contact Information)

Lab SOP Number	Title, Revision Date, and Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM	Modified for Project Work? (Y/N)
CA-604	Acid Digestion of Aqueous Samples by USEPA Method 3010 for ICP Analysis of Total or Dissolved Metals, 04/10, Revision 5.	Definitive	Groundwater / TAL Metals	Not applicable (digestion)	No Variance	N
CA-608	Trace Metals Analysis By ICP-AES Using USEPA Method 6010, 04/12, Revision 13.	Definitive	Groundwater / TAL Metals	Inductively Coupled Plasma (ICP) – Atomic Emission Spectroscopy (AES)	No Variance	N
CA-772	Colorimetric Analysis of Hexavalent Chromium Using the Automated Konelab Multiwavelength Photometric Analyzer, 02/13, Revision 03.	Definitive	Groundwater / Hexavalent Chromium	Spectrophotometer	No Variance	N
CA-627	Trace Metals Analysis By ICP-MS Using USEPA Method 6020, 04/12, Revision 8.	Definitive	Groundwater / TAL Metals	ICP-MS	No Variance	N
CA-719	Total Dissolved Solids (Filterable Residue) by USEPA Method 160.1 and Standard Methods 2540 C, 05/12, Revision 7.	Screening	Groundwater / Total Dissolved Solids	Balance	No Variance	N

Lab SOP Number	Title, Revision Date, and Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM	Modified for Project Work? (Y/N)
CA-739	Titrimetric Determination of Total Alkalinity by USEPA Method 310.1 and SM 2320 B using the Mettler Dl25 Autotitrator, and Calculation of the Component Forms of Alkalinity by SM 4500–$C0_2$ D, 05/12, Revision 9.	Screening	Groundwater / Alkalinity	Autotitrator	No Variance	N
CA-742	Anions by Ion Chromatography (IC) – Method 300.0 and SW-846 9056, 06/12, Revision 9.	Screening	Groundwater / Anions	Ion Chromatograph	No Variance	N
CA-763	Analysis of TOC, DOC, and TIC in Aqueous Samples using the Shimadzu Carbon Analyzer: USEPA Method 415.1, SW846 9060 and SM5310B, 05/12, Revision 7.	Screening	Groundwater / Total Organic Carbon	Total Organic Carbon Analyzer	No Variance	N

Notes:

1. Lab Accreditation and Certification requirements and verification for the work of this project are presented on Worksheet #30.

SAP Worksheet #24: Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
ICP-AES – Metals	ICAL	At the beginning of each day or if QC is out of criteria.	One point calibration plus a blank per manufacturer's guidelines.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-608
	ICV	Once after each ICAL, prior to beginning a sample run.	%R must be within 90-110% for all project compounds.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	Calibration Blank (CB)	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst, Department Manager	
	CCV	After every 10 samples and at the end of each run sequence.	%R must be within 90-110% for all project compounds.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
	Low-level Calibration Check Standard (if using one-point ICAL)	Daily after one-point ICAL.	%R must within 80%-120% for all project compounds.	Correct problem, then reanalyze.	Analyst, Department Manager	
	ICS - ICSA & ICSB	Daily, before sample injections	ICSA recoveries must be less than the absolute value of the LOD and ICSB %Rs must be within 80-120%.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Department Manager	CA-608

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
ICP-MS Metals	Tune	Daily prior to calibration.	Mass calibration must be within 0.1 atomic mass unit (amu) from the true value. Resolution must be <0.9 amu full width at 10% peak height. Four injections %RSD must be <5%.	Perform necessary equipment maintenance.	Analyst, Department Manager	CA-627
ICP-MS Metals (continued)	ICAL - 4 point calibration plus blank	Daily prior to sample analysis.	Correlation coefficient (r) must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	
	ICV	Once after each ICAL, and before beginning a sample run.	%R must be within 90-110% for all project compounds.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	CCB	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence. For negative blanks, absolute value < LOD.	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	
	CCV	After every 10 samples and at the end of each run sequence.	%R must be within 90-110% for all project compounds.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
	Low-level calibration check standard	Daily after ICAL	Within ± 20% of true value.	Correct problem, then reanalyze.	Analyst, Department Manager	
	ICS - ICSA & ICSB	Daily, before sample injections	ICSA recoveries must be less than the absolute value of the LOD(2) and ICSB %Rs must be within 80-120%.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Konelab –Ammonia, Hexavalent Chromium	ICAL - 5 points plus a calibration blank	Daily ICAL prior to sample analysis	Correlation coefficient (r) must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-770, CA-772
	ICV	Once after each ICAL, prior to beginning a sample run.	Ammonia: %R must be within 90-110%. Hexavalent Chromium: The %R must be within 90-110% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	CCV	Beginning and end of each run sequence and every 10 samples.	%R must be within 90-110%	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
Autotitrator / Alkalinity	CCV	One after every 10 samples	%R must within 80%-120%	(1) If the CCV fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.	Analyst, Department Manager	CA-739
Balance / Total Dissolved Solids	Balance Verification	Every day before use.	0.0020 ± 0.0002 0.1000 ± 0.0005 1.0000 ± 0.0005 10.0000 ± 0.0005 50.0000 ± 0.0005 100.0000 ± 0.0005	Investigate problem. Do not use balance until verification has passed.	Analyst, Department Manager	CA-719
Total Organic Carbon Analyzer / Total Organic Carbon	ICAL – Minimum of a 5-point calibration curve plus a blank is prepared.	Initially, when the daily CCV does not pass, but, no longer than every 3 months.	Correlation coefficient ≤ 0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Department Manager	CA-763
	ICV	Once after each ICAL, prior to beginning a sample run.	%R must within 80%-120%	If the ICV fails high, report samples that are <PQL. Redigest, recalibrate and/or reanalyze other samples.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Total Organic Carbon Analyzer / Total Organic Carbon (continued)	CCV	Every 10 samples and at the end of the run	%R must within 80%-120%	If the CCV fails high, report samples that are <PQL. Recalibrate and/or reanalyze samples back to last acceptable CCV recovery.	Analyst, Department Manager	
Ion Chromatography / Chloride, Sulfate, Nitrite, Nitrate	ICAL – A minimum of a 5-point calibration is prepared.	Prior to sample analysis.	R must be ≥0.995.	Correct problem and rerun calibration.	Analyst, Department Manager	CA-742
	ICV	Once after each ICAL prior to sample analysis.	The %R must be within 90-110% of true value and retention times (RTs) must be within appropriate windows.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	CCV	After every 10 samples and at the end of the sequence.	The %R must be within 90-110% of true value and all project analytes must be within established RT windows.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	

SAP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	TAL Metals, Simultaneously Extracted Metals	Torch, nebulizer chamber, pump, pump tubing.	Prior to ICAL and as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-608
ICP-MS	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	TAL Metals	Torch, nebulizer, spray chamber, pump tubing.	Prior to ICAL and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-627
Konelab	Check and clean segments weekly, clean reagent tubes monthly. Change lamp, change diluent and wash tubes, change mixing paddles and syringes, change dispensing needle, all as needed.	Ammonia, Hexavalent Chromium	Reagent tubes, lamp, wash tubes, paddles, syringes, dispensing needles.	Prior to initial calibration and/or as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-770, CA-772
Autotitrator	Fill pH electrode as needed, clean stirring paddle weekly, fill rinse as needed.	Alkalinity	pH electrode, stirring paddle, reagent bottles.	As necessary	Acceptable CCV	Correct the problem reanalyze CCV.	Analyst, Department Manager	CA-739

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Balance	Weights	Total Dissolved Solids	Cleanliness	Prior to sample weighing	Acceptable CCV	Correct the problem, reanalyze CCV.	Analyst, Department Manager	CA-719
TOC Combustion Analyzer	Check level of dilution water, drain vessel water, humidifier water, auto sampler rinse water and phosphoric acid vessel and fill as needed. Replace oxygen cylinder.	Total Organic Carbon	Tubing, sample boat, syringe, humidifier, rinse reservoir, phosphoric acid vessel, oxygen pressure	Prior to initial calibration and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-763
Ion Chromatograph	Check regenerate pump tubing and replace as needed. Clean or regenerate column as needed. Replace analytical column or guard column as needed. Change suppressor as needed.	Chloride, Sulfate, Nitrite, Nitrate	Tubing, column, suppressor.	Prior to initial calibration and/or as necessary.	Passing ICAL or CCV.	Correct problem and repeat calibration or CCV.	Analyst, Department Manager	CA-742

SAP Worksheet #26: Sample Handling System

(UFP-QAPP Manual Appendix A)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT	
Sample Collection (Personnel/Organization):	Resolution Field Team Staff
Sample Packaging (Personnel/Organization):	Resolution Field Team Staff
Coordination of Shipment (Personnel/Organization):	Resolution Field Team Staff (Refer to Worksheet #30 for the contact information for each laboratory providing analytical services)
Type of Shipment/Carrier:	Air Courier (FedEx Overnight) or Laboratory Courier
SAMPLE RECEIPT AND ANALYSIS	
Sample Receipt (Personnel/Organization):	Assigned laboratory personnel (Refer to Worksheet #30 for laboratories providing analytical services)
Sample Custody and Storage (Personnel/Organization):	Assigned laboratory personnel (Refer to Worksheet #30 for laboratories providing analytical services)
Sample Preparation (Personnel/Organization):	Assigned laboratory personnel (Refer to Worksheet #30 for laboratories providing analytical services)
Sample Determinative Analysis (Personnel/Organization):	Assigned laboratory personnel (Refer to Worksheet #30 for laboratories providing analytical services)
SAMPLE ARCHIVING	
Field Sample Storage (No. of days from sample collection):	None anticipated. All samples will be sent to the laboratory.
Sample Extract/Digestate Storage (No. of days from extraction/digestion):	Sample extracts and digests will be archived by the laboratory for 180 days from sample receipt.
SAMPLE DISPOSAL	
Personnel/Organization:	Assigned laboratory personnel (Refer to Worksheet #30 for laboratories providing analytical services)
Number of Days from Analysis:	Unused sample will be archived by the laboratories for 180 days from sample receipt.

SAP Worksheet #27: Sample Custody Requirements

(UFP-QAPP Manual Section 3.3.3)

Sample Labeling

Samples will be labeled in accordance with Resolution *Procedure 3-03, Recordkeeping, Sample Labeling, and Chain-of-Custody*, presented in Appendix B. Effort will be made to label the sample containers prior to collection with a subset of information that is predetermined (e.g., method, preservative), and complete the sample labels immediately after collection (e.g., sample collection date, sampler's initials).

Sample Nomenclature

Samples will be identified using a unique sample identification system. Sample nomenclature will be based on the sample locations presented in Worksheet #18, and will adhere to the following scheme:

Field Samples

Field samples will be named using the following conventions. The backslashes in the descriptions are included to separate each section of the description but will not be included in the sample identification.

Groundwater: Site Identifier/Sample Type/Sample Location Number/Event Code

Site Identifier
Site Identifier will be:
CRF – Coddington Cove Rubble Fill Area

Sample Type
Sample type will consist of two characters:
MW – Monitoring Well

Sample Location Number
Sample location numbers that will be used are those presented on Worksheet #18 exclusive of the two-letter description for sample type. For example, sample location MW01 is sample location number 01.

Event Code
The event code will consist of the month, day and year in the following format:
01Jan1900

Field QC Samples

Field QC samples will be named using the following convention. The backslashes in the descriptions are included to separate each section of the description but will not be included in the sample identification.

QC Type / Date / Number

QC Type
QC type will consist of two characters:
RB – Rinsate (equipment) Blank
FD – Field Duplicate
Matrix spike/matrix spike duplicates and matrix duplicates will be noted on the chains of custody, but since they consist of only additional volume for a field sample, no distinct nomenclature is required.

Date
Date will consist of six numbers for MMDDYY.

Number
Number will consist of two digits. Each sample type will be numbered sequentially on a daily basis.

Conventions for Naming Sampling Locations

Future monitoring wells will be numbered consecutively beginning with MW09.

Sample Custody Procedures

Sample custody will be maintained in accordance with Resolution *Procedures 3-03, Recordkeeping, Sample Labeling, and Chain-of-Custody* and *3-04, Sample Handling, Storage, and Shipping*, both presented in Appendix B.

Samples will be collected by and remain in the custody of Resolution until they are either released to the air courier that will be used for shipment to the laboratory or transferred to the custody of the laboratory as represented by the laboratory courier. A sample is considered to be in someone's custody if it:

- Is in his/her possession
- Is in his/her view, after being in his/her possession
- Is in his/her possession and has been placed in a secured location
- Is in a designated secure area

Sample custody procedures followed by each subcontract laboratory are presented in the laboratory quality manuals and standard operating procedures.

Chains-of-Custody and Sample Login Forms

Resolution will use hardcopy chains of custody available from the fixed-laboratories performing the analyses. Examples of the chain of custody form, sample login form, and internal chain of custody form that will be used are presented in Figures 27-1 through 27-3.

Figure 27-1
Chain of Custody for Katahdin Analytical Services

Katahdin ANALYTICAL SERVICES
600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400
Fax: (207) 775-4029

Chain of Custody

Client:	Contact:	Phone #: ( )	Fax #: ( )
Address:	City:	State:	Zip Code:
Purchase Order #:	Proj. Name/No.:		Katahdin Quote #:
Bill (if different than above):		Address:	
Sampler (Print/Sign):			Copies To:

LAB USE ONLY	Work Order #: Katahdin Project Number
Remarks:	
Shipping Info:	FEDEX UPS CLIENT
Airbill No:	
Temp C	Temp Blank Intact Not Intact

*	Sample Description	Date/Time Collected	Matrix	No. of Containers	Analysis and Container Type Preservatives: Filt. Y / N	Filt. Y / N	Filt. Y / N	Filt. Y / N	Filt. Y / N	Filt. Y / N	Filt. Y / N	Filt. Y / N	Filt. Y / N	Filt. Y / N

COMMENTS:

Relinquished By:	Date/Time	Received By:	Relinquished By:	Date/Time	Received By:
Relinquished By:	Date/Time	Received By:	Relinquished By:	Date/Time	Received By:

The terms and conditions on the following page hereof shall govern services, except when a signed contractual agreement exists.

Figure 27-2
Katahdin Analytical Services Login Form

Katahdin Analytical Services, Inc. **Sample Receipt Condition Report**

Client:	KAS PM:	Sampled By:
Project:	KIMS Entry By:	Delivered By:
KAS Work Order#:	KIMS Review By:	Received By:
SDG #:	Cooler: _____ of _____	Date/Time Rec.:

Receipt Criteria	Y	N	EX*	NA	Comments and/or Resolution
1. Custody seals present / intact?					
2. Chain of Custody present in cooler?					
3. Chain of Custody signed by client?					
4. Chain of Custody matches samples?					
5. Temperature Blanks present? If not, take temperature of any sample w/ IR gun.					Temp (°C):
Samples received at <6 °C w/o freezing?					Note: Not required for metals analysis.
Ice packs or ice present?					The lack of ice or ice packs (i.e. no attempt to begin cooling process) may not meet certain regulatory requirements and may invalidate certain data.
If temp. out, has the cooling process begun (i.e. ice or packs present) and sample collection times <6hrs., but samples are not yet cool?					Note: No cooling process required for metals analysis.
6. Volatiles free of headspace: **Aqueous:** No bubble larger than a pea **Soil/Sediment:**					
Received in airtight container?					
Received in methanol?					
Methanol covering soil?					
7. Trip Blank present in cooler?					
8. Proper sample containers and volume?					
9. Samples within hold time upon receipt?					
10. Aqueous samples properly preserved? Metals, COD, NH3, TKN, O/G, phenol, TPO4, N+N, TOC, DRO, TPH – pH <2					
Sulfide - >9					
Cyanide – pH >12					

* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments

QA-048 – Revision 1 – 04/16/2010

Figure 27-3
Katahdin Internal Chain of Custody

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services
Container History Report (ct08)
11-JUN-12 09:38 AM

Container ID / Sample	Type	Status	Amount	Transaction Date	From Response	From Location	From Operator	To Response	To Location	To Operator	Hazard Code	Dispose Code	Dispose Date	Comments
SF3170-1A														
	250mL Plastic+HNO3	INTACT		01-JUN-12 08:54:09	CUSTODY	WALK-IN	NTILLOTSON	METALS PREP	METALS PREP	NTILLOTSON				
	250mL Plastic+HNO3	INTACT		31-MAY-12 12:38:51	LOGIN	LOGIN	DDOYON	CUSTODY	WALK-IN	DDOYON				
	250mL Plastic+HNO3	INTACT		31-MAY-12 12:20:01	LOGIN	LOGIN	SBROWN	LOGIN	LOGIN	SBROWN				

SAP Worksheet #28-1: Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater

Analytical Group: ICP-MS Metals

Analytical Method/ SOP Reference: SW846 6020A / CA-627

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples of similar matrix.	No target metals > ½ LOQ (> LOQ for common laboratory contaminants) and > 1/10 the amount measured in any sample or 1/10 the PAL, whichever is greater. For negative blanks, absolute value < LOD. Blank result must not otherwise affect sample results (see DoD QSM Box D-1).	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per digestion batch of 20 or fewer samples of similar matrix.	%R must be within 80-120%, allowing for the marginal exceedances presented in DoD QSM Table G-1.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS	One per digestion batch or SDG or every 20 samples.	%R should be within 80-120%if sample < 4x spike added.	Flag results for affected analytes for all associated samples with an"N."	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits for LCS.
Post-digestion Spike	When dilution test fails or analyte concentration in all samples < 50x LOD	%R should be within 75-125%.	Run associated samples by method of standard addition or flag results.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Laboratory Duplicate	One per digestion batch or SDG or every 20 samples.	Project-specific criteria: If values are ≥ 5x LOQ, RPD should be ≤ 20%. If values are < 5x LOQ, Absolute Difference should be ≤ LOQ.	Flag results for affected analytes for all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution	One per preparation batch of twenty or fewer samples of similar matrix.	If original sample result is at least 50x LOQ, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with "E."	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
IS	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte	For each sample, IS intensity must be within 30-120% of that of initial calibration standard.	Reanalyze affected samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Per the method, for each sample, IS intensity must be ≥ 70% of that of initial calibration standard.

SAP Worksheet #28-2: Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater

Analytical Group: ICP-AES Metals

Analytical Method/ SOP Reference: SW846 6010C / CA-608

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples of similar matrix.	No target metals> ½ LOQ (> LOQ for common laboratory contaminants) and > 1/10 the amount measured in any sample or 1/10 the PAL, whichever is greater. For negative blanks, absolute value must be < LOD. Blank result must not otherwise affect sample results (see DoD QSM Box D-1).	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per digestion batch of 20 or fewer samples of similar matrix (varies by lot).	%R must be within DoD QSM limits, allowing for the marginal exceedances presented in DoD QSM Table G-1.	Re-digest and reanalyze all associated samples for affected analyte.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Matrix Spike	One per digestion batch or SDG or every 20 samples.	%R should be within the DoD QSM limits for LCS, if sample < 4x spike added.	Flag results for affected analytes for all associated samples with "N."	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits for LCS.
Post-digestion Spike	When dilution test fails or analyte concentration in all samples < 50x LOD	%R should be within 75-125%.	Run associated samples by method of standard addition or flag results.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Laboratory Duplicate	One per digestion batch or SDG or every 20 samples.	Project-specific criteria: If values are ≥ 5x LOQ, RPD should be ≤ 20%. If values are < 5x LOQ, Absolute Difference should be ≤ LOQ.	Flag results for affected analytes for all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution	One per preparation batch of 20 or fewer samples of similar matrix.	If original sample result is at least 50x LOQ, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with "E."	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

SAP Worksheet #28-3: Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater

Analytical Group: Hexavalent Chromium

Analytical Method/ SOP Reference: SW846 7196A / CA-772

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per prep batch	No analytes detected > 1/2 LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. See Box D-1 of DoD QSM v 4.1.	Investigate source of contamination. If blank value > LOQ report sample results if < PQL or > 10 x the blank value. Otherwise re analyze	Analyst, Supervisor	Accuracy / Bias / Contamination	Same as SOP QC Acceptance Limits.
LCS	One per prep batch	85-115 %R	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G of DoD QSM). If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst, Supervisor	Accuracy / Bias	Same as SOP QC Acceptance Limits.
MS	One for every sample matrix analyzed	85-115 % Recovery	Narrate	Analyst, Supervisor	Accuracy / Bias	Same as SOP QC Acceptance Limits.

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Laboratory Duplicate	One per digestion batch or SDG or every 20 samples.	Project-specific criteria: If values are ≥ 5x LOQ, RPD should be ≤ 20%. If values are < 5x LOQ, Absolute Difference should be ≤ LOQ.	Investigate problem and reanalyze sample in duplicate. If RPD still >20, report original result with notation or narration. Flag results for affected analytes for all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ	NA	Analyst, Supervisor	Accuracy	Same as QC Acceptance Limits.

SAP Worksheet #28-4: Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater

Analytical Group: Alkalinity

Analytical Method/ SOP Reference: SM2320B / CA-739

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the PAL, whichever is greater. Blank result must not otherwise affect sample results (see DoD QSM Box D-1).	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS	One for every set 10 samples	%R must be within: 75-125	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 if both results are >3 x LOQ; RPD ≤100% for samples ≤ 3 x LOQ	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet #28-5: Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater

Analytical Group: Anions (Chloride, Nitrate, Nitrite, Sulfate)

Analytical Method/ SOP Reference: SW846 9056A / CA-742

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the PAL, whichever is greater. Blank result must not otherwise affect sample results (see DoD QSM Box D-1).	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G of DoD QSM v4.2).	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One set for every set 20 samples	%R must be within: 80-120 RPD ≤ 15% (between MS and MSD).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/Bias	RPD ≤ 15%; Recovery same as Method/SOP QC Acceptance Limits for LCS.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ. Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	Correct problem and reanalyze sample and duplicate.	Analyst, Laboratory Department Manager, and Data Validator	Precision	RPD < 20%

SAP Worksheet #28-6: Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater

Analytical Group: Ammonia

Analytical Method/ SOP Reference: EPA 350.1 / CA-770

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the PAL, whichever is greater. Blank result must not otherwise affect sample results (see DoD QSM Box D-1).	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G of DoD QSM v4.2).	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One set for every set 20 samples	%R must be within: 80-120 RPD ≤ 15% (between MS and MSD).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/Bias	RPD ≤ 15%; Recovery same as Method/SOP QC Acceptance Limits for LCS.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ. Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	Correct problem and reanalyze sample and duplicate.	Analyst, Laboratory Department Manager, and Data Validator	Precision	RPD < 20%

SAP Worksheet #28-7: Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater

Analytical Group: Total Organic Carbon (Method 9060)

Analytical Method/ SOP Reference: SW846 9060 / CA-763

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the PAL, whichever is greater. Blank result must not otherwise affect sample results (see DoD QSM Box D-1).	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS	One for every set 10 samples	%R must be within: 75-125	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 if both results are >3 x LOQ; RPD ≤100% for samples ≤ 3 x LOQ	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet #28-8: Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater

Analytical Group: Total Dissolved Solids

Analytical Method/ SOP Reference: SM 2540C / CA-719

QC Sample:	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > LOQ and > 1/10 the amount measured in any sample or 1/10 the PAL, whichever is greater. Blank result must not otherwise affect sample results (see DoD QSM Box D-1).	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 if both results are >3 x LOQ; RPD ≤100% for samples ≤ 3 x LOQ	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	RPD < 20%

SAP Worksheet #28: Notes

(UFP-QAPP Manual Section 3.4)

Calc. – calculated
CAS – Chemical Abstracts Service
DL – Detection Limit
DoD QSM – Department of Defense Quality Systems Manual
DQI – Data Quality Indicator
ICAL – Initial Calibration
ICP – Inductively Coupled Plasma
IDL – Instrument Detection Limit
IS – Internal Standard
LCS – Laboratory Control Spike
LCSD – Laboratory Control Spike Duplicate
LOD – Limit of Detection
LOQ – Limit of Quantitation
MS – Matrix Spike
MSD – Matrix Spike Duplicate
NA – Not Applicable
PAL – Project Action Limit
QA – Quality Assurance
QC – Quality Control
R – Recovery
RPD – Relative Percent Difference
Rev. – Revision
SDG – Sample Delivery Group
SOP – Standard Operating Procedure

SAP Worksheet #29: Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document [1]	Where Maintained
Field Logbooks	Resolution Office/SharePoint
Chain-of-Custodies	Resolution Office/SharePoint
Equipment Calibration Records	Resolution Office/SharePoint
Field Parameter Collection Records	Resolution Office/SharePoint
Electronic Data Deliverables	Resolution SharePoint; data will be loaded in project database; data will be loaded into the Navy's NIRIS system.
Data Validation Reports	Resolution Office/SharePoint; revised data will be updated in project database.

Notes:
(1) – A Tech Memo will be prepared for formal documentation of the information inputs, analytical data, and data evaluation for this investigation program. The final Tech Memo will be maintained in the Resolution Office/Share Point files, will be uploaded to the Navy's NIRIS system and will be placed in the Administrative Record.

SAP Worksheet #30: Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix/ Investigation	Analytical Group	Sample Locations/ID Numbers	Analytical SOP	Deliverable Level[1]	Data Package Turnaround Time	Laboratory/Organization (name and address, contact person and telephone number)[2]	Backup Laboratory/Organization (name and address, contact person and telephone number)
Groundwater Investigation							
	ICP-MS Metals	See attached figures and Worksheet #27	CA-627	Full	21 calendar days	Katahdin Analytical Services[3]	NA
	ICP-AES Metals	See attached figures and Worksheet #27	CA-604, CA-608	Full	21 calendar days	Katahdin Analytical Services[3]	NA
	Hexavalent Chromium	See attached figures and Worksheet #27	CA-772	Full	21 calendar days	Katahdin Analytical Services[3]	NA
Geochemical/ Biological Indicator Groundwater Characterization[5]							
	Alkalinity	See attached figures and Worksheet #27	CA-739	Full	21 calendar days	Katahdin Analytical Services[3]	NA
	Anions (Chloride, Nitrate, Nitrite, Sulfate)	See attached figures and Worksheet #27	CA-742	Full	21 calendar days	Katahdin Analytical Services[3]	NA
	Total Organic Carbon (Method 9060M)	See attached figures and Worksheet #27	CA-763	Full	21 calendar days	Katahdin Analytical Services[3]	NA
	Ammonia	See attached figures and Worksheet #27	CA-770	Full	21 calendar days	Katahdin Analytical Services[3]	NA
	Total Dissolved Solids	See attached figures and Worksheet #27	CA-719	Full	21 calendar days	Katahdin Analytical Services[4]	NA

Note 1: DATA DELIVERABLES

Full

A full data deliverable is the most complete deliverable available from commercial laboratories. The full data deliverable consists of a hardcopy data deliverable (HDD) and Electronic Data Deliverable (EDD).

The detailed requirements for a full data deliverable HDD are presented in the Resolution's *Statement Of Work To Master Services Agreement For Laboratory Analytical Services, Appendix C* (February 2012). The requirements are consistent with the requirements set forth in Section 5.10 of the DoD QSM, V4.2. The HDD will be delivered in paper and/or pdf. The pdf must be bookmarked and searchable, and will be submitted on CD ROM to the Project Data Validation Coordinator or Project Chemist (to be determined prior to data receipt). The HDD will consist of the following:

I. Cover Sheet
II. Table of Contents
III. Case Narrative
IV. Laboratory Communications
V. Sample Management Records (COC's, shipping documents, sample condition receipt, etc.)
VI. Method Sections
    A. Summary Forms
    B. Supporting Raw Data (including copies of pertinent logbooks and bench sheets)

The EDD will be delivered on CD ROM or via email to the Project Data Validation Coordinator or Project Chemist (to be determined prior to data receipt). Detailed EDD requirements are presented in the *Resolution Data Deliverables Specification for EQuIS Deliverables* (March 16, 2010), presented in the work order issued to the laboratory. The EDD is an Resolution Custom EQuIS 4-file format which can be obtained directly from the EQuIS Data Processor. Each EDD will comprise 4 files, to describe samples, tests, results, and batches. The format file has two different sections for samples, Field and Lab, only one of which can be included in the EDD. The list of valid values applicable to the project and the section (Field or Lab) to use will be communicated by the Resolution Data Manager at project setup, prior to submission of samples.

Field Worksheets

Documents associated with these analyses will consist of the worksheets used for low-flow groundwater sampling and instrument calibration. Water quality parameters measured during low-flow sampling of groundwater wells (DO, ORP, specific conductance, temperature, and turbidity) and ferrous iron test kit results will be recorded on the Groundwater Sample Collection Record Form presented in Resolution Consultant's *Procedure 3-14, Monitoring Well Sampling*. The associated instrument calibration and calibration checks for water quality parameters will be documented on the Field Instrument Calibration Form presented in Resolution's *Procedure 3-24, Water Quality Parameter Testing for Groundwater Sampling*. The calibration and calibration checks for the ferrous iron test kits will be recorded in the field logbook. Resolution's Procedures are provided in Appendix B. Completed worksheets will be provided along with copies of the field logbook pages by the Resolution's Field Team Leader to the Resolution CTO Manager within one week of the conclusion of each field sampling event.

Note 2: LABORATORY CONTACT INFORMATION

Katahdin Analytical Services
600 Technology Way
Scarborough, Maine 04074

Project Manager: Jennifer Obrin
Federal Programs Project Manager
Direct: 207.874.2400 x17
Fax: 207.775.4029
Email: jobrin@katahdinlab.com

(Note that a laboratory courier may be available)

Note 3: DoD ELAP ACCREDITATION

3. The laboratory holds current DoD ELAP Accreditation for this analysis. Certificates of accreditation are presented in Appendix C: Katahdin Analytical Services Certificate Number L2223 is granted through February 1, 2016. Current accreditation will be verified prior to submission of samples for analysis. Katahdin Analytical Services also holds current certification from the State of Rhode Island and Providence Plantations Department of Health for Potable and Non-potable Water Organic and Inorganic Chemistry (Certificate Number LAO00303, current through 12/30/2013).

ABBREVIATIONS:
NA- Not Available
RC – Resolution Consultants

SAP Worksheet #31: Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing CA (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Safety Audit	None planned.	N/A	N/A	N/A	N/A	N/A	N/A
On-Site Technical Audit of Field Activities	None planned.	N/A	N/A	N/A	N/A	N/A	N/A

Notes:
CA - corrective action

SAP Worksheet #32: Assessment Findings and Corrective Action Responses Table

(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Safety Audit	None planned.	N/A	N/A	N/A	N/A	N/A
On-Site Technical Audit of Field Activities	None planned.	N/A	N/A	N/A	N/A	N/A

SAP Worksheet #33: Quality Assurance Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Technical Audit of Field Activities: Audit Report	None planned.	N/A	N/A	N/A
Safety Audit	None planned.	N/A	N/A	N/A
Daily Field Progress Reports	Daily	Daily	Resolution Field Team Leader	Resolution CTO Manager
Data Validation Reports	One per SDG; after laboratory data is received and validated	Estimated 30 days after receipt of complete SDG	Resolution Data Validation Coordinator	Resolution CTO Manager, Resolution Project Chemist
Data Usability Report	Once per field program; after all data are validated	Estimated 15 days after completion of data validation	Resolution Project Chemist	Resolution CTO Manager
Tech Memo	Once per report iterate	Estimated 24 days after completion of data validation	Resolution CTO Manager	Navy, USEPA, RIDEM

Notes:
CTO - Contract Task Order
RI – Remedial Investigation
QA - Quality Assurance
SDG – Sample Delivery Group
RI Report includes Human Health and Ecological Risk Assessments

SAP Worksheets #34-36: Data Verification and Validation (Steps I and IIa/IIb) Process Table

(UFP-QAPP Manual Section 5.2.1), (UFP-QAPP Manual Section 5.2.2), (Figure 37 UFP-QAPP Manual), (Table 9 UFP-QAPP Manual)

Data Review Input	Description	Responsible for Verification (name, organization)	Step I / IIa / IIb [1]	Internal/ External
Chain-of-Custody Forms	The Resolution Field Team Leader or designee will review and sign each chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The chain-of-custody forms will be signed by the sampler and a copy will be retained for the project file, the Resolution CTO Manager, and the Resolution Data Validator. See SOP.	Field Team Leader and Field Crew, Resolution	I	Internal
Chain-of-Custody Forms	The Laboratory Sample Custodian will review the sample shipment for completeness and integrity and will sign accepting the shipment.	Laboratory Sample Custodian	I	External
Chain-of-Custody Forms	The data validator will check that the chain-of-custody form was signed and dated by the Resolution SS or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses. The data validator will confirm that the custody and integrity of the samples were maintained from collection to analysis and that custody records are complete and any deviations are recorded.	Data Validator, Resolution	I	Internal
Field SOPs/Field Logs/Sample Collection	Confirm that all sampling SOPs were followed. Verify appropriate records of calibration. Verify that deviations have been documented and performance criteria have been achieved, that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken chain of custody from sample collection to report generation. Verify that the correct sampling and analytical procedures were applied. Verify that the SAP was followed as written and that any deviations are documented.	CTO Manager, Field Team Leader, or designee, Resolution	IIa	Internal
Sample Tables	Verify that proposed samples have been collected.	Field Team Leader and Field Crew, Resolution	IIa	Internal

Data Review Input	Description	Responsible for Verification (name, organization)	Step I / IIa / IIb [1]	Internal/ External
Sample Log Sheets	Log sheets completed as samples are collected in the field are verified for completeness and are maintained at the project office.	CTO Manager, Field Team Leader, or designee, Resolution	IIa	Internal
Field QC Samples	Verify that field QC samples listed in Worksheet #12 were collected as required.	Field Team Leader or designee, Resolution	IIa	Internal
Sample Coordinates	Sample locations will be validated to be correct and in accordance with the SAP (compare map of proposed locations to map of actual locations).	CTO Manager, Field Team Leader, or designee, Resolution	IIa	Internal
Analytical SOPs	Confirm that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Laboratory Quality Assurance Manager	IIa	Internal
Documentation of Method QC Results	Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the Laboratory Quality Assurance Manager will contact Resolution for guidance prior to report preparation.	Laboratory Quality Assurance Manager	IIa	Internal
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory Quality Assurance Manager will sign the case narrative for each data package.	Laboratory Quality Assurance Manager	IIa	Internal
Analytical Data Packages	Verify that the data package contains all the elements required by the laboratory Master Services Agreement and laboratory work order. Missing information will be requested from the laboratory, and data validation (if applicable) will be suspended until missing data are received.	Data Validator, Resolution	IIa	External
Documentation of Analytical Reports for Completeness	Confirm that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as described below per Worksheet #36. Verify all data have been transferred correctly and completely to the final SQL database.	Data Validator, Resolution	IIa	External

Data Review Input	Description	Responsible for Verification (name, organization)	Step I / IIa / IIb [1]	Internal/ External
Electronic Data Deliverables	The electronic data will be compared to the chain-of-custody form and hard copy data package to verify accuracy and completeness.	Data Validator, Resolution	IIa	External
Analytical Data Packages	For the matrices and analyses presented in the table below*, Limited Data Validation will be performed using criteria for the methods listed in Worksheet #'s 12, 15, 24, and 28, the DoD QSM, laboratory work order, and laboratory SOPs. If not addressed in the worksheets or DoD QSM, the logic outlined in the *Region I, USEPA-New England Data Validation Functional Guidelines For Evaluating Environmental Analyses* (December 1996), *USEPA National Functional Guidelines for Superfund Organic Methods Data Review* (June 2008), and *USEPA National Functional Guidelines for Inorganic Methods Data Review* (January 2010) will be used to apply qualifiers to data.	Data Validator, Resolution	IIb	External

Data Review Input	Description	Responsible for Verification (name, organization)	Step I / IIa / IIb [1]	Internal/ External
Analytical Data Packages (in addition to Limited Validation)	Data Results: Verify that the summary form results match the raw data. Holding Times: Verify that the samples were shipped and stored at the required temperature and that the sample pH values for chemically preserved samples meet the requirements listed in Worksheet # 19. Verify that the analyses were performed within the holding times listed in Worksheet # 19. Laboratory Data Results for Accuracy: Confirm that the laboratory QC samples listed in Worksheet # 28 were analyzed and that the MPCs listed in Worksheet # 12 were met for all field samples and QC analyses. Verify that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met. Field and Laboratory Duplicate Analyses for Precision: Verify the field sampling precision by checking the RPD for field duplicate samples. Verify laboratory precision by checking RPDs or %D values from laboratory duplicate, MS/MSD, MS/Sample Duplicate, and LCS/LCSD analyses. Confirm compliance with the methods and project MPC accuracy goals listed in Worksheet # 12 and 28. Project Quantitation Limits for Sensitivity: Verify that the QLs listed in Worksheet # 15 were achieved. Sample Results for Representativeness: Verify that the laboratory recorded the temperature of each sample at sample receipt and the pH of chemically preserved samples to confirm sample integrity from sample collection to analysis.	Data Validator, Resolution	IIa/IIb	External
Data Validation Report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all data qualifications. Print a copy of the project database, qualified data depicting data qualifiers, and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	Data Validator, Resolution	IIa/IIb	External

Data Review Input	Description	Responsible for Verification (name, organization)	Step I / IIa / IIb [1]	Internal/ External
Project Action Limits	Discuss the impact of matrix interferences or sample dilutions performed, because of the high concentration of one or more contaminants, on the other target compounds reported as not detected. Document this usability issue and inform the Resolution CTO Manager.	Data Validator, Resolution	IIa/IIb	External
SAP QC Sample Documentation	Verify that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Verify that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the Laboratory Quality Assurance Manager shall have contacted the Resolution Project Chemist or CTO Manager.	Data Validator, Resolution	IIa/IIb	External
Analytical Data Deviations	Determine the impact of any deviation from sampling or analytical methods, SOP requirements, and matrix interferences on the analytical results.	Data Validator, Resolution	IIa/IIb	External

Notes: 1 IIa=compliance with methods, procedures, and contracts [see Table 10, page 117, UFP-QAPP manual, V.1, March 2005.]
IIb=comparison with measurement performance criteria in the SAP [see Table 11, page 118, UFP-QAPP manual, V.1, March 2005]

***Data Undergoing Limited Data Validation**	
Matrix	**Analytical Group**
Groundwater	ICP-MS Metals;
	ICP-AES Metals
	Hexavalent Chromium
	Alkalinity
	Anions (Chloride, Nitrate, Nitrite, Sulfate)
	Ammonia

SAP Worksheet #37: Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used

Resolution Consultant's data validation staff will validate all laboratory data in accordance with the protocols described in Worksheets #34-36. These procedures are consistent with USEPA National and Regional Data Validation Functional Guidelines. The Project Chemist, in conjunction with the project team, will determine whether the analytical data meet the requirements to support the RI. The results of laboratory measurements will be compared to the data quality objectives described in Worksheet #11.

Describe the evaluative procedures used to assess overall measurement error associated with the project

During the data validation process, the validator will use information confirming sample identification, sample preparation, analysis within holding time, instrument calibration data, and results of QC samples designed to assess blank contamination, analytical precision, and accuracy to identify any limitations in data use and, if known, data bias. The validator will apply qualifiers as needed to reflect any limitations on the use of specific data points and prepare a report detailing the information reviewed, data limitations, and overall usability. Patterns of data use limitations or anomalies which become apparent during the validation process or during use will be reviewed with the Project Chemist and the appropriate laboratory. Data that do not meet the quality acceptance limits of Worksheet #28, or quality levels of Worksheet #15, or analytical performance criteria specified in Worksheet #12 will be clearly identified in the database so data users are aware of any limitations associated with data usability. Details of the problems identified during data validation and the bias in the data will be provided in the associated validation memorandum.

Identify the personnel responsible for performing the usability assessment

Data validation will be performed by Resolution's data validation staff under the supervision of the Data Validation Coordinator and Project Chemist.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies

The documentation generated during data validation will include a comprehensive memorandum that describes the information reviewed, the results of this review, and provides a recommendation

on overall data usability and limitations on specific data points. The memorandum and associated validation worksheets provide information on the samples included in the review and the date they were collected, the condition of samples when received at the laboratory and any discrepancies noted during the receiving process, verification of sample preparation and analysis within the method specified holding time, instrument calibration information, review of associated QC analyses including blanks, LCSs, MSs, and field and/or laboratory duplicates, and verification of selected reported values from raw data. As a result of this review standard qualifiers are entered into the database so that data users can readily identify any limitations associated with a specific data point.

References

RIDEM, 2010. Rules and Regulations for Groundwater Quality. State of Rhode Island and Providence Plantations Department of Environmental Management, Office of Water Resources. June 2010.

RIDEM, 2011. Rules and Regulations for the Investigation and Remediation of Hazardous Materials Releases, State of Rhode Island and Providence Plantations Department of Environmental Management, Office of Waste Management. March 31, 1993 as amended November 2011.

Tetra Tech, April, 2005. Results of Field Sampling Effort – May and July 2004, Coddington Cove Rubble Fill Area, SA-04, Naval Station Newport, Newport, Rhode Island.

Tetra Tech, November 2010. Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan), Study Area Screening Evaluation, Site 04, Coddington Cove Rubble Fill Area, NAVSTA Newport, Newport, Rhode Island.

Tetra Tech NUS, Inc., May 2012. Revised Draft Study Area Screening Investigation Report, Site 04 Coddington Cove Rubble Fill Area, NAVSTA, Newport, Rhode Island. May 2012.

USEPA, 1996. Low Stress (Low-flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells. USEPA, Region 1. Revision 2. July 30, 1996.

USEPA, 2002. Guidance for Quality Assurance Project Plans, USEPA QA/G-5. December 2002.

USEPA, 2005. Uniform Federal Policy for Quality Assurance Project Plans. Version 1. March 2005.

USEPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process, USEPA QA/G-4. February 2006.

Figures

RESOLUTION CONSULTANTS

Drawn:	JB	03/12/2013
Approved:	MC	03/12/2013
Project #:	60268621	

Map Location

N

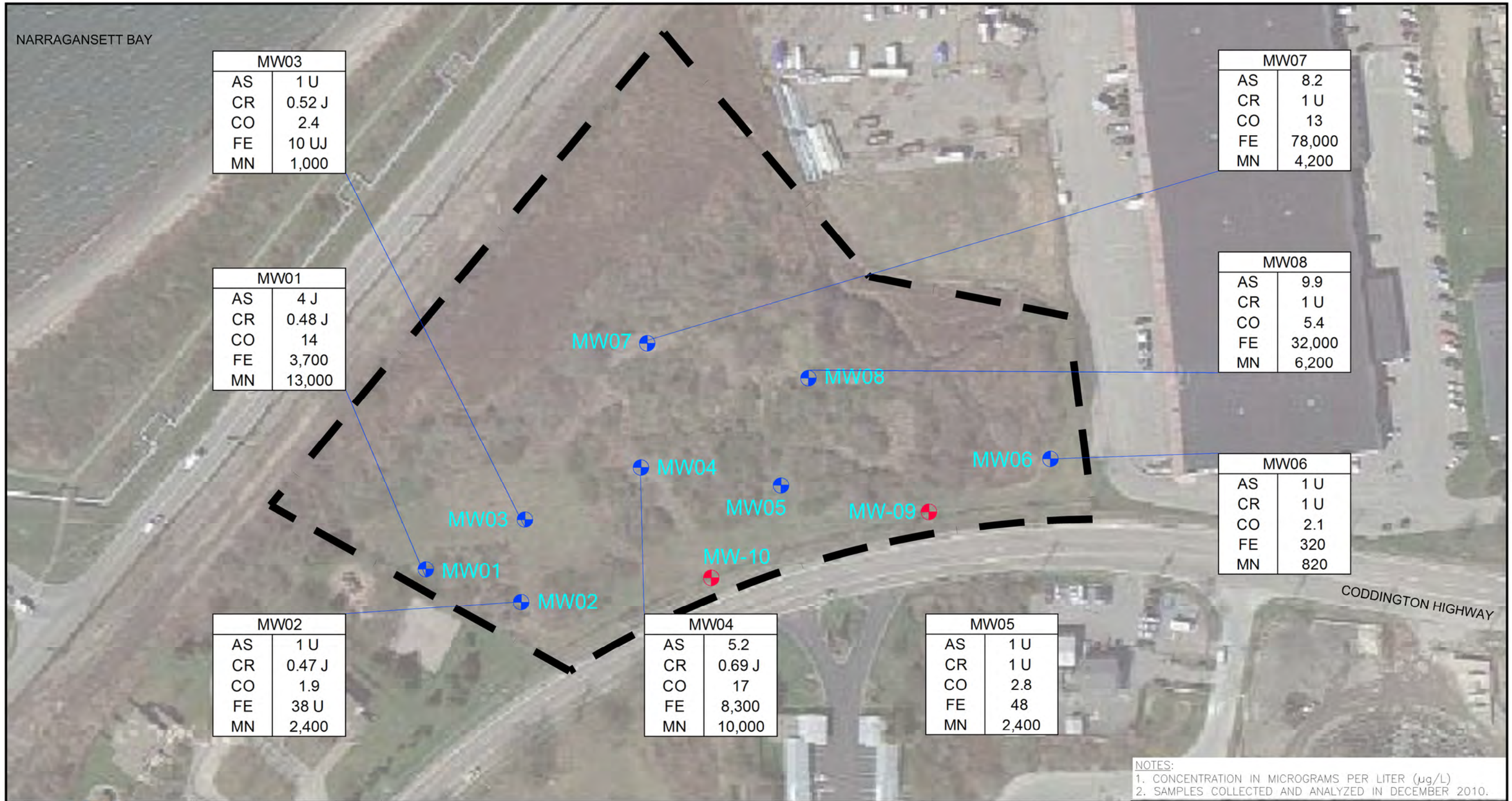
0 2,000 4,000 8,000

Scale in Feet

FIGURE 1
REGIONAL LOCATION

CODDINGTON COVE RUBBLE FILL AREA
NAVSTA NEWPORT, RHODE ISLAND

G:\Projects\HAZWASTE\Newport - Coddington\Maps\Figure 1 - Regional Location.mxd



NOTES:
1. CONCENTRATION IN MICROGRAMS PER LITER (µg/L)
2. SAMPLES COLLECTED AND ANALYZED IN DECEMBER 2010.

RESOLUTION CONSULTANTS

Drawn:	BCV	09/19/2013
Approved:	J.M.	09/19/2013
Project #:	60268621	

Map Location

LEGEND

WELL-ID EXISTING MONITORING WELL

WELL-ID PROPOSED MONITORING WELL

PROPERTY BOUNDARY

MW__	MONITORING WELL ID
AS	ARSENIC
CR	CHROMIUM
CO	COBALT
FE	IRON
MN	MANGANESE
U	UNDETECTED
J	ESTIMATED

N

50 0 100

GRAPHIC SCALE IN FEET*

FIGURE - 2
SITE PLAN, GROUNDWATER MONITORING RESULTS (METALS) AND PROPOSED MONITORING WELL LOCATIONS

CODDINGTON COVE
NEWPORT, RHODE ISLAND

File: C:\PLOTS\NewPort-Coddington\Drawing1alt.dwg Layout: GW-RESULTS Then: VERSION8 Plotted: Sep 20, 2013 - 11:09am Xrefs:

NOTES:
1. CROSS SECTIONS ARE ON FIGURE 4.

NARRAGANSETT BAY

B

SW04
SD04

SW05
SD05

SD03

MW07

TP13

SW07
SD07

MW08

SW06
SD06

TP14

A'

SW02
SD02

MW04

MW06

SW01
SD01

TP12

MW03

MW05

TP15

A

MW01

MW02

B'

CODDINGTON HIGHWAY

RESOLUTION CONSULTANTS

Drawn:	BCV	09/19/2013
Approved:	J.M.	09/19/2013
Project #:		60268621

Map Location

LEGEND

- WELL-ID MONITORING WELL
- SW_ SURFACE WATER (SW) SEDIMENT (SD) SAMPLELOCATION
- SD_
- TP_ TEST PIT SAMPLE LOCATION
- PROPERTY BOUNDARY
- B — B' CROSS SECTION LOCATION

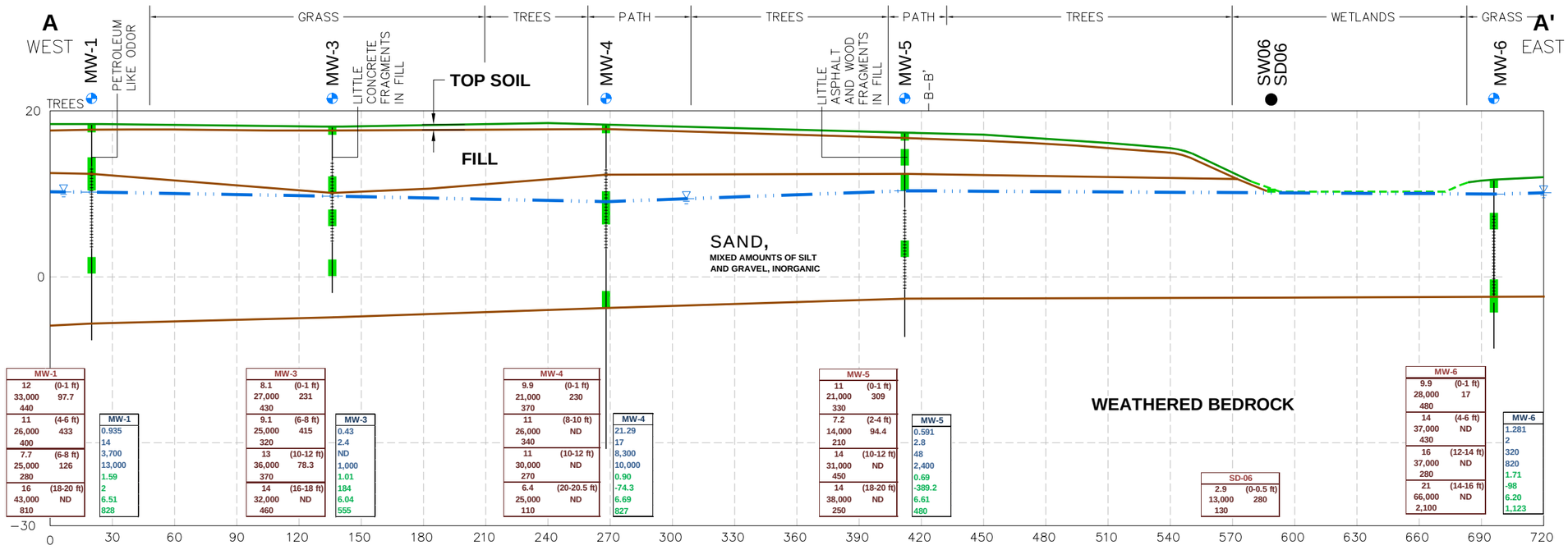
N

50 0 100

GRAPHIC SCALE IN FEET*

FIGURE - 3
GEOLOGIC CROSS SECTION
LOCATION PLAN
SECTIONS A-A' & B-B'

CODDINGTON COVE
NEWPORT, RHODE ISLAND



NOTE:

1. SEE FIGURE 3 FOR CROSS SECTION LOCATION PLAN.
2. TPH RESULT FOR MW-6 (0-1) FROM DRO RESULTS.

LEGEND

LOCATION-ID
COBALT (DEPTH/FT)
IRON TPH
MANGANESE
Soil (MG/KG)

SOIL DATA KEY

LOCATION-ID
Groundwater (UG/L)
VOCs+SVOCs
Cobalt
Iron
Manganese
Field Parameters
DO (MG/L)
ORP (MV)
pH (UNIT LESS)
SPECIFIC CONDUCTANCE uS/cm

GROUNDWATER DATA KEY

GROUNDWATER TABLE (DASHED WHERE INFERED)

GROUNDSURFACE (DASHED WHERE INFERED)

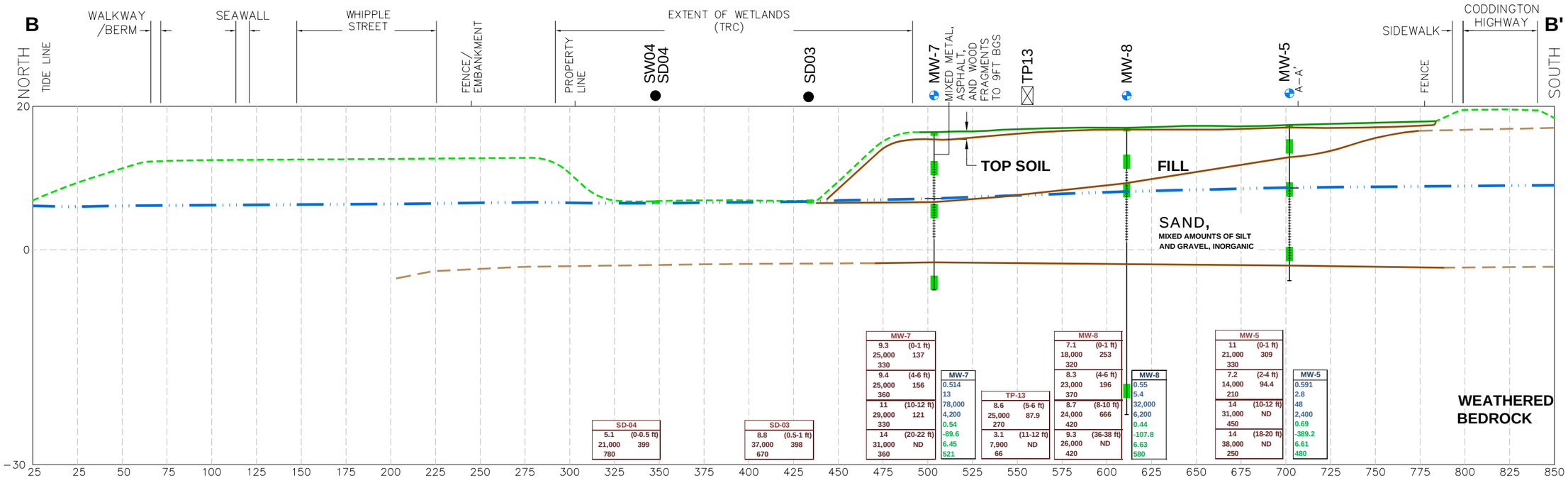
STRATOGRAPHIC CONTACT (DASHED WHERE INFERRED)

WELL RISER (SOLID PIPE)

WELL SCREEN (SLOTTED PIPE)

SOIL SAMPLE INTERVAL

DO	DISSOLVED OXYGEN
ORP	OXIDATION REDUCTION POTENTIAL
VOC	VOLATILE ORGANIC COMPOUND
SVOC	SEMIVOLATILE ORGANIC COMPOUND
TPH	TOTAL PETROLEUM HYDROCARBONS



RESOLUTION CONSULTANTS

FIGURE 4

CROSS SECTIONS A-A' & B-B'

CODDINGTON COVE
NEWPORT, RI

60268621

DRWN: BcV/C-MA

DATE: 10/30/2013

File: G:\Projects\Navy CLEAN AECOM-EnSafe JV\Newport\CTO WE19 - CORR\CADD_ACM-60268621_C-01.dwg Layout: A-A User: vershonb Plotted: Oct 30, 2013 - 4:53pm Xref's:

Appendix A

November 2010 Final Sampling and Analysis Plan

N62661.AR.002394
NS NEWPORT
5090.3a

FINAL SAMPLING AND ANALYSIS PLAN STUDY AREA SCREENING EVALUATION FOR SITE 4 CODDINGTON RUBBLE FILL AREA NS NEWPORT RI

11/01/2010

TETRA TECH NUS

SAP Worksheet #1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
November 2010

Study Area Screening Evaluation
Site 04, Coddington Cove Rubble Fill Area
NAVSTA Newport
Newport, Rhode Island

Prepared for:
Naval Facilities Engineering Command, Mid Atlantic
9742 Maryland Avenue
Norfolk, Virginia 23511-3095

Prepared by:
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, Pennsylvania 19046-1433
(610) 491-9688

Prepared under:
Contract Number N62470-08D-1001
“CLEAN” Contract Task Order No. WE48

QAPP Worksheet #1 (UFP-QAPP Manual Section 2.1) -- Approval Page

Document Title: "Sampling and Analysis Plan, Site 04, Coddington Cove Rubble Fill Area, NAVSTA Newport, Newport, Rhode Island", April 2010

Lead Organization: Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic

Preparer's Name and Organizational Affiliation: Tetra Tech NUS, Inc.

Preparer's Address and Telephone Number: 234 Mall Boulevard Suite 260, King of Prussia, Pennsylvania 19046-1433

Preparation Date (Month/Year): April 2010

Investigative Organization's Project Manager:

Tom A. Campbell 4/27/10

Signature/Date
Tom Campbell, Tetra Tech

Investigative Organization's Project Quality Assurance (QA) Manager:

T. Johnston 4-26-10

Signature/Date
Tom Johnston PhD, Tetra Tech.

Lead Organization's Project Manager:

JOHNSON.WINOMA.A.1229628980

Digitally signed by JOHNSON.WINOMA.A.1229628980
DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=JOHNSON.WINOMA.A.1229628980
Date: 2010.04.27 13:09:34 -04'00'

Signature/Date
Winoma Johnson, NAVFAC

Lead Organization QA Officer:

Kenneth Bo 4/27/10

Signature/Date
Government Chemist

EXECUTIVE SUMMARY

This Sampling and Analysis Plan (SAP) has been prepared to support a Site Assessment Screening Evaluation (SASE) and human health and ecological screening risk assessments of the Coddington Cove Rubble Fill (CCRF) Area (Installation Restoration Site 04) at Naval Station (NAVSTA) Newport, Rhode Island. It presents field sampling and laboratory analytical methods and procedures for soil, groundwater, surface water and sediment that have been potentially impacted by fill materials placed at the CCRF Area (the Site).

Site 04, the CCRF Area, is the location of a former fill/disposal area on an approximately 8 acre vacant lot located at Naval Station (NAVSTA) Newport. The site consists of a now vegetated former fill area and a wetland area and is located on the northwest side of Coddington Highway. The boundary between Newport and Middletown, Rhode Island crosses through the site in a southeast-northwest direction.

Railroad tracks, the Defense Highway, and the former Derecktor Shipyard bound the site to the northwest, beyond which are Coddington Cove and Narragansett Bay. To the north and east are a fenced, secure storage area, an open storage area, and NAVSTA Newport Building 47. A Navy housing development abuts the site to the southwest. A chain link fence encloses the site along all boundaries.

Limited information is available regarding the historical land use of Site 04. According to a 1983 Initial Assessment Study (IAS), from 1978 to 1982 the site was a disposal area for inert rubble materials including concrete, asphalt, metal, slate, wood, brush, and a small amount of ash (Envirodyne, 1983). Reviewed records did not indicate additional land use practices.

The central portion of the site consists of the former disposal area with an abutting wetland, with site topography sloping downward from the disposal area into the wetland. Historic aerial photographs indicate two possible fill/disposal areas on Site 04. The fill/disposal areas appear to extend into the onsite wetland area. A stream which forms much of the north-northeastern boundary of the wetland flows west and north before exiting the site at the northernmost portion of the site.

A Preliminary Assessment (PA) was conducted at the site in May and June 2004. Analytical results from soil and groundwater samples (“direct dip” samples of groundwater that had infiltrated into test pits) that were collected from test pits indicated the presence of contaminants, including volatile organic compounds (VOCs) at low concentrations, polycyclic aromatic hydrocarbons (PAHs), pesticides, polychlorinated biphenyls (PCBs), diesel-range organics (DRO), and metals. Based on these results, the PA concluded that the site be carried forward to the Site Assessment Screening Evaluation (SASE) step, in accordance with the Federal Facilities Agreement (FFA) for NAVSTA Newport Installation Restoration (IR) Program sites.

Sampling is proposed as part of this SASE to address media that have not yet been evaluated, including groundwater, surface water, and sediment. In addition, the former disposal area will be further characterized with regard to nature and extent of contaminants. Data collected as part of this effort will be used to complete human health and ecological screening risk assessments.

SAP Worksheets

SAP Worksheet #1 -- Title And Approval Page 1
SAP Worksheet #2 -- Sap Identifying Information 9
SAP Worksheet #3 -- Distribution List 12
SAP Worksheet #4 -- Project Personnel Sign-Off Sheet 13
SAP Worksheet #5 -- Project Organizational Chart 16
SAP Worksheet #6 -- Communication Pathways 17
SAP Worksheet #7 -- Personnel Responsibilities And Qualifications Table 18
SAP Worksheet #8 -- Special Personnel Training Requirements Table 20
SAP Worksheet #9a -- Project Scoping Session Participants Sheet 21
SAP Worksheet #9b -- Project Scoping Session Participants Sheet 22
SAP Worksheet #9c -- Project Scoping Session Participants Sheet 23
SAP Worksheet #9d -- Project Scoping Session Participants Sheet 24
SAP Worksheet #9e -- Project Scoping Session Participants Sheet 26
SAP Worksheet #9f -- Project Scoping Session Participants Sheet 27
SAP Worksheet #10 – Conceptual Site Model 28
SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements 34
SAP Worksheet #12 -- Measurement Performance Criteria Table 39
SAP Worksheet #13 -- Secondary Data Criteria And Limitations Table 40
SAP Worksheet #14 -- Summary Of Project Tasks 41
SAP Worksheet #15a -- Groundwater Reference Limits And Evaluation Table 51
SAP Worksheet #15b – Surface Water Reference Limits And Evaluation Table 59
SAP Worksheet #15c – Soil Reference Limits And Evaluation Table 65
SAP Worksheet #15d – Sediment Reference Limits And Evaluation Table 71
SAP Worksheet #16 -- Project Schedule / Timeline Table (Optional Format) 77
SAP Worksheet #17 -- Sampling Design And Rationale 78
SAP Worksheet #18 -- Sampling Locations And Methods/Sop Requirements Table 83
SAP Worksheet #19 -- Analytical Sop Requirements Table 84
SAP Worksheet #20 -- Field Quality Control Sample Summary Table 87
SAP Worksheet #21 -- Project Sampling Sop References Table 89
SAP Worksheet #22 -- Field Equipment Calibration, Maintenance, Testing, And Inspection Table 91
SAP Worksheet #23 -- Analytical Sop References Table 92
SAP Worksheet #24 -- Analytical Instrument Calibration Table 97
SAP Worksheet #25 -- Analytical Instrument And Equipment Maintenance, Testing, And Inspection Table 107
SAP Worksheet #28 – Laboratory Qc Samples Table 115
SAP Worksheet #26 – Sample Handling System 110
SAP Worksheet #27 – Sample Custody Requirements Table 111
SAP Worksheet #28 – Laboratory Qc Samples Table 115
SAP Worksheet #29 -- Project Documents And Records Table 127
SAP Worksheet #30 -- Analytical Services Table 128
SAP Worksheet #31 -- Planned Project Assessments Table 129
SAP Worksheet #32 -- Assessment Findings And Corrective Action Responses 130
SAP Worksheet #33 -- Qa Management Reports Table 131
SAP Worksheet #34 -- Verification (Step I) Process Table 132
SAP Worksheet #35 -- Validation (Steps Iia And Iib) Process Table 133
SAP Worksheet #36 -- Analytical Data Validation (Steps Iia And Iib) Summary Table 135
SAP Worksheet #37 -- Usability Assessment 137

List of Tables

17-1 Summary of Proposed Borings and Monitoring Wells
17-2 Summary of Proposed Test Pits
17-3 Summary of Proposed Sediment and Surface Water Samples

List of Figures

10-1 Site Locus
10-2 2005 PA Investigation – Sample Location Map
10-3 Conceptual Site Model
11-1 Navy Ecological Risk Assessment Tiered Approach
17-1 Proposed Sample Location Map

REFERENCES

APPENDICES

A Tetra Tech and USEPA SOPs
B Field Documentation Forms
C Project-Specific Field Task Procedures
  C-1 Project-Specific Procedures for Water Level Measurement and Groundwater Sampling
  C-2 Project-Specific Soil Sampling Procedures
D Analytical Specification
E Analytical SOPs and Certifications
F PA Investigation Analytical Summary Tables and Data Validation Memo
G PA Investigation Test Pit Log Sheets
H Historic Aerial Photographs

Acronyms

AA	Atomic Absorption
ASTM	American Society for Standards and Materials
CA	Corrective Action
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CLP	Contract Laboratory Program
COC	Contaminant of Concern
CRDL	Contract-Required Detection Limit
CSM	Conceptual Site Model
CTO	Contract Task Order
CWA	Clean Water Act
DoD	Department of Defense
DCN	Document Control Number
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
EPA	Environmental Protection Agency
FCR	Field Change Request
FFA	Federal Facilities Agreement
FOL	Field Operations Leader
FS	Feasibility Study
FSP	Field Sampling Plan
FTMR	Field Task Modification Request
GC	Gas Chromatograph
GC/MS	Gas Chromatograph/Mass Spectrometer
GIS	Geographic Information System
GPC	Gel Permeation Chromatography
GPS	Global Positioning System
GW	Ground Water
HASP	Health and Safety Plan
HSM	Health and Safety Manager
ICP	Inductively Coupled Plasma
IDQTF	Intergovernmental Data Quality Task Force
IR	Installation Restoration
LCS	Laboratory Control Sample
LFB	Laboratory Fortified Blank
LIMS	Laboratory Information Management Systems
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols (Manual)
MARSSIM	Multi-Agency Radiation Survey and Site Investigation Manual
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MOU	Memorandum of Understanding
MPC	Measurement Performance Criteria
MQO	Measurement Quality Objectives
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MSR	Management Systems Review
NEIC	National Enforcement Investigations Center
NIST	National Institute of Standards and Technology
NPDES	National Pollutant Discharge Elimination System
NPL	National Priorities List
PA/SI	Preliminary Assessment/Site Investigation
PAHs	Polycyclic Aromatic Hydrocarbons
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PCBs	Polychlorinated Biphenyls

PDF	Portable Document Format
PG	Professional Geologist
PM	Project Manager
PQOs	Project Quality Objectives
PRP	Potentially Responsible Party
PRQL	Project-Required Quantitation Limit
PT	Proficiency Testing (previously known as performance evaluation (PE) sample)
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
QMP	Quality Management Plan
QS	Quality System
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RTC	Response to Comments
SAP	Sampling and Analysis Plan
SD	Standard Deviation
SDG	Sample Delivery Group
SDWA	Safe Drinking Water Act
SOP	Standard Operating Procedure
SQLs	Sample Quantitation Limits
SRM	Standard Reference Material
SSO	Site Safety Officer
SVOA	Semivolatile Organic Analytes
SVOC	Semivolatile Organic Compounds
SW	Surface Water
TCLP	Toxicity Characteristic Leaching Procedure
TBD	To Be Determined
TSA	Technical Systems Audit
UFP	Uniform Federal Policy
VOA	Volatile Organic Analytes
VOC	Volatile Organic Compounds

SAP Worksheet #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: Site 04, Coddington Cove Rubble Fill Area, NAVSTA Newport
Operable Unit: NA
Contractor Name: Tetra Tech NUS, Inc. (Tetra Tech)
Contract Number: N62470-08D-1001
Contract Title: Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic Comprehensive Long-Term Environmental Action Navy (CLEAN)
Work Assignment Number (optional): Contract Task Order (CTO) WE48

1. This SAP was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (U.S. EPA 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (U.S. EPA 2002)* .

2. Identify regulatory program: National Contingency Plan (NCP); Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA)

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
NAVSTA Newport RPM Meeting	September 16, 2009
EPA email correspondence	October 22, 2009
RIDEM email correspondence	December 10, 2009

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
Technical Memorandum, Record Review and Field Sampling Plan for Coddington Cove Rubble Fill Area (Study Area 04)	May 2004

6. List organizational partners (stakeholders) and connection with lead organization:

U. S. Environmental Protection Agency (USEPA), Region I – lead regulatory agency
Rhode Island Department of Environmental Management (RIDEM) – regulatory agency
Naval Station Newport – property holder

---

7. Lead organization (see WS 7 for detailed list of data users)

NAVFAC Mid-Atlantic

8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

Not applicable

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	N/A
2	Table of Contents SAP Identifying Information	N/A
3	Distribution List	N/A
4	Project Personnel Sign-Off Sheet	N/A
Project Organization		
5	Project Organizational Chart	N/A
6	Communication Pathways	N/A
7	Personnel Responsibilities and Qualifications Table	N/A
8	Special Personnel Training Requirements Table	N/A
Project Planning/ Problem Definition		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	N/A
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	N/A
11	Site-Specific Project Quality Objectives	N/A
12	Measurement Performance Criteria Table	N/A
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	N/A
14	Summary of Project Tasks	N/A
15	Reference Limits and Evaluation Table	N/A
16	Project Schedule/Timeline Table	N/A
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	N/A
18	Sampling Locations and Methods/ SOP Requirements Table Sample Location Map(s)	N/A
19	Analytical Methods/SOP Requirements Table	N/A
20	Field Quality Control Sample Summary Table	N/A
21	Project Sampling SOP References Table Sampling SOPs	N/A
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	N/A
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	N/A
24	Analytical Instrument Calibration Table	N/A
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	N/A
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	N/A
27	Sample Custody Requirements, Procedures/SOPs Sample Container	N/A

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
	Identification Example Chain-of-Custody Form and Seal	
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	N/A
Data Management Tasks		
29	Project Documents and Records Table	N/A
30	Analytical Services Table Analytical and Data Management SOPs	N/A
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	N/A
32	Assessment Findings and Corrective Action Responses Table	N/A
33	QA Management Reports Table	N/A
D. Data Review		
34	Verification (Step I) Process Table	N/A
35	Validation (Steps IIa and IIb) Process Table	N/A
36	Validation (Steps IIa and IIb) Summary Table	N/A
37	Usability Assessment	N/A

SAP Worksheet #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients [1]	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Ed Corack, PE	Remedial Project Manager, NAVFAC	NAVFAC Mid-Atlantic	757-341-1985	edward.corack@navy.mil	NA
Cornelia Mueller	IR Program Manager	NAVSTA Newport Environmental	401-841-7561	cornelia.mueller@navy.mil	NA
Jonathan Tucker	Project Chemist	NAVFAC Atlantic	757-322-8288	Jonathan.tucker@navy.mil	NA
Dave Barclift	Ecological Risk Assessor	NAVFAC	215-897-4913	David.barclift@navy.mil	NA
Bonnie Capito	Librarian	NAVFAC Atlantic Librarian		Bonnie.capito@navy.mil	NA
Ginny Lombardo	Remedial Project Manager	USEPA Region 1 Federal Facilities	617-918-1754	Lombardo.Ginny@epamail.epa.gov	NA
Gary Jablonski	Remedial Project Manager	RIDEM Div Site Remediation	401-222-2797 ext 7148	Gary.jablonski@dem.ri.gov	NA
Tom Campbell	Project Manager	Tetra Tech	978-474-8404	Thomas.campbell@tetratech.com	NA
Thomas Johnston	Quality Assurance Manager	Tetra Tech	412-921-8615	tom.johnston@tetratech.com	NA
Walt Roudebush	Lab PM	Trimatrix Laboratories	616-975-4500	komarp@trimatrixlabs.com	NA
TBD[2]	TBD	Drilling Subcontractor	TBD	TBD	NA

1 Each person in this table will be responsible for distributing copies of this SAP to appropriate personnel within their organization. For example, Tom Campbell (PM) will be responsible for distributing copies of this SAP to all project personnel listed in Worksheet #4 (Project Personnel Sign-Off Sheet).

2 Drilling subcontractor information will be entered into worksheet #3 after the drilling subcontract is awarded in August/September 2010.

SAP Worksheet #4 -- Project Personnel Sign-Off Sheet
(UFP-QAPP Manual Section 2.3.2)

Key personnel will be instructed to read the SAP prior to attending an internal site-specific kick-off meeting for field activities. The Tetra Tech PM will track when the reviews have been completed, obtain signatures, and ensure that the completed sign-off sheet is included in the central project file.

Organization: Tetra Tech

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Tom Campbell	Tetra Tech PM and Project Health and Safety Officer (SSO)/Oversees project execution	978-474-8400		All	
Mike Horton	Tetra Tech Field Operations Leader (FOL) and Site Safety Officer (SSO)/Oversees field project and execution and provides safety oversight	978-474-8400		All	
Lucy Guzman	Tetra Tech /Project Chemist/Oversees chemistry portions of SAP development and implementation, including DV operations	978-474-8400		All	
Tom Johnston	Tetra Tech /Quality Assurance Manager (QAM)/Oversees SAP preparation and reviews	412-921-8615		All	
Matt Soltis	Tetra Tech /Health and Safety Manager (HSM)/Oversees HASP preparation	412-921-8912		HASP	
Gary Glennon	Tetra Tech /Project Data Manager/Oversees tracking and control of electronic data	978-474-8414		Worksheets #12, #14, #15, #19, #20, #23-28, #30, #34-37	

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Walt Roudebush	Trimatrix Lab PM/Oversees execution of lab analyses	616-975-4500		Worksheets #12, #14, #15, #19, #20, #23-28, #30, #34-36	
Aaron Bernhardt	Tetra Tech/Ecological Risk Assessor, ecological risk assessment/Oversees Ecological Risk Assessment process	412-921-8433		Worksheets #10, #11, #14, #15, #17, #18, #19	
Lee Ann Sinagoga	Tetra Tech/Human Health Risk Assessor/Oversees Human Health Risk Assessment process	412-921-8887		Worksheets #10, #11, #14, #15, #17, #18, #19	
To Be Determined: Project Manager will assign	Field Sample Collection Specialist/Sample collection, packaging, shipment	978-474-8400			
To Be Determined: Project Manager will assign	GIS specialist, rendering of spatial data	978-474-8400			
To Be Determined: Project Manager will assign	Database specialist, tabular data presentation and analysis	978-474-8400			

Organization: Navy

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Ed Corack, PE	Project Manager, prime contractor oversight and management/Oversees	757-341-1985		All	

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
	project execution				
Cornelia Mueller	IR Program Manager, overall lead agency responsible for project performance/Oversees project execution	401-841-7561		All	
David Barclift	Ecological Risk Assessor/Oversees project risk assessment	215-897-4913			

Organization: RIDEM

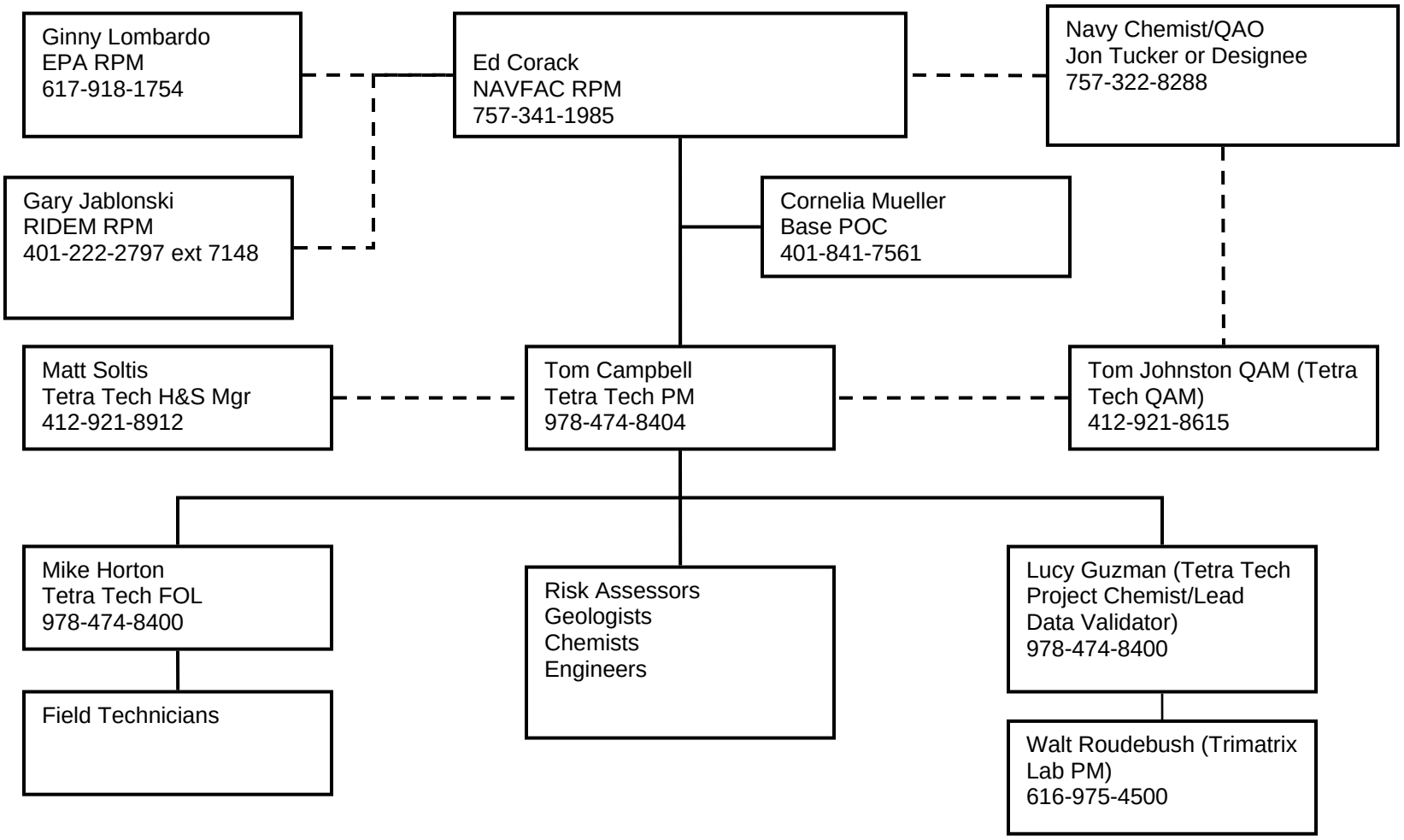
Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Gary Jablonski	Project Manager, oversight of Navy performance/Oversees project execution	401-222-2797 ext 7148		All	

Organization: U.S. EPA Region I

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Ginny Lombardo	Project Manager, oversight of Navy performance/Oversees project execution	617-918-1754		All	

SAP Worksheet #5 -- Project Organizational Chart
(UFP-QAPP Manual Section 2.4.1)

Lines of Authority ————
Lines of Communication ·····



SAP Worksheet #6 -- Communication Pathways
(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure (timing, pathway to & from, etc.)
SAP amendments	Tetra Tech PM NAVFAC RPM	Tom Campbell Ed Corack	978-474-8400 757-341-1985	After realizing an amendment is needed, PM will send NAVFAC a concurrence letter within 7 days. The NAVFAC RPM will sign the letter within 5 days of receipt.
Schedule changes	Tetra Tech PM	Tom Campbell	978-474-8400	When impact is realized, send NAVFAC a schedule concurrence letter within 7 days or prior to the first affected deliverable date.
Field issues that require changes in scope or implementation of field work	Tetra Tech FOL NAVFAC RPM Tetra Tech PM	Mike Horton Ed Corack Tom Campbell	978-474-8400 757-341-1985 978-474-8400	FOL informs PM the day the issue is realized. PM informs the NAVFAC RPM of the issue within 1 day of the FOL's notification. PM also sends a concurrence letter to the NAVFAC within 7 days, if project scope is affected. The NAVFAC RPM will sign the letter within 5 days of receipt. Document changes on a Field Task Modification Request (FTMR) form.
Stop work recommendations, for example, to protect workers from unsafe conditions/situations or to prevent a degradation in laboratory quality of work	Tetra Tech FOL/SSO Tetra Tech PM Tetra Tech QAM Laboratory PM Tetra Tech Project Chemist	Mike Horton Tom Campbell Dr. Tom Johnston Phil Komar Lucy Guzman	978-474-8400 978-474-8400 412-921-8615 616-846-9528 978-474-8400	On the day the problem is identified, FOL, SSO, or QAM informs PM and documents problem on a FTMR. On the same day of the notification from the FOL, PM informs the NAVFAC and Installation RPMs. When issue is related to chemical data, Laboratory PM notifies Project Chemist within 2 days of identification of problem. Project Chemist notifies Tetra Tech PM within 1 day.

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Ed Corack	RPM	NAVFAC Midlant	Oversees project implementation, including scoping, data review, and evaluation.
Gary Jablonski	RPM	RIDEM	Participates in scoping, data review, evaluation, and approves the SAP.
Cornelia Mueller	Site Manager	NAVSTA Newport POC	Oversees site activities and participates in scoping, data review, evaluation, and reviews the SAP.
Ginny Lombardo	RPM	USEPA Region 1	Participates in scoping, data review, and evaluation.
Tom Campbell	PM	Tetra Tech	Oversees and manages project technical operations, finances, and schedule. Coordinates Tetra Tech activities with NAVFAC RPM and other members of Project Team. Manages subcontractors.
Mike Horton	FOL	Tetra Tech	Supervises, coordinates, and performs field sampling activities**.**
TBD	SSO	Tetra Tech	The SSO will be responsible for training and monitoring site conditions.
Dr. Tom Johnston	QAM	Tetra Tech	Reviews the SAP, oversees preparation of lab scope, and data quality review. Ensures quality aspects of the CLEAN program are implemented.
Matt Soltis	HSM	Tetra Tech	Oversees the Tetra Tech CLEAN Health and Safety Program.
Lucy Guzman	Project Chemist	Tetra Tech	Provide chemistry support to the project including oversight of laboratory procurement and performance:

Name	Title/Role	Organizational Affiliation	Responsibilities
Gary Glennon	Project Data Manager	Tetra Tech	Oversees the tracking and control of electronic data.
Walt Roudebush	Laboratory PM	Trimatrix Laboratories	Coordinates analyses with lab chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker will be required to have completed a 40-hour course (and 8-hour refresher, if applicable) in Health and Safety Training as described under Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120(b)(4). Safety requirements are addressed in greater detail in the site specific Health and Safety Plan (HASP), prepared under separate cover.

SAP Worksheet #9a -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Site 04, Coddington Cove Rubble Fill Area Projected Date(s) of Sampling: TBD Project Manager: Tom Campbell	Site Name: Site 04, Coddington Cove Rubble Fill Area Site Location: NAVSTA Newport, Rhode Island

Date of Session: September 16, 2009
Scoping Session Purpose: NAVSTA Newport RPM Meeting

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Winoma Johnson	RPM	Navy	(757) 444-0825	winoma.johnson@navy.mil	Navy RPM
Paul Kulpa	State RPM	RIDEM	(401) 222-2797 ext. 7111	paul.kulpa@dem.ri.gov	State RPM
Ginny Lombardo	RPM	USEPA Region 1	617-918-1754	Lombardo.Ginny@epamail.epa.gov	Federal RPM
Cornelia Mueller	NAVSTA Newport Base Contact	Navy	401-841-7561	cornelia.mueller@navy.mil	Base Contact
Stephen Parker	Base Coordinator	Tetra Tech	978-474-8400	Stephen.parker@tetratech.com	Base coordinator

Comments/Decisions: An agenda topic for the September 2009 NAVSTA Newport RPM meeting was the initiation of a UFP QAPP for a SASE at the Coddington Cove Rubble Fill Area site. Tetra Tech distributed the conceptual site model and a site plan figure of the Coddington Cove Rubble Fill Area site at the meeting. Stephen Parker requested that input be received by email from EPA and RIDEM identifying what data points they view to be important for the SASE investigation. In addition, Tetra Tech requested from EPA and RIDEM what data endpoints are needed for this study area and what is the basis for that need.

Action Items: Email response from EPA and RIDEM to explain technical positions and sampling endpoints in support of SASE.

Consensus Decisions: None.

SAP Worksheet #9b -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Site 04, Coddington Cove Rubble Fill Area Projected Date(s) of Sampling: TBD Project Manager: Tom Campbell	Site Name: Site 04, Coddington Cove Rubble Fill Area Site Location: NAVSTA Newport, Rhode Island

Date of Session: October 22, 2009
Scoping Session Purpose: Email Correspondence from EPA

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Winoma Johnson	RPM	Navy	(757) 444-0825	winoma.johnson@navy.mil	Navy RPM
Paul Kulpa	State RPM	RIDEM	(401) 222-2797 ext. 7111	paul.kulpa@dem.ri.gov	State RPM
Ginny Lombardo	RPM	USEPA Region 1	617-918-1754	Lombardo.Ginny@epamail.epa.gov	Federal RPM
Cornelia Mueller	NAVSTA Newport Base Contact	Navy	401-841-7561	cornelia.mueller@navy.mil	Base Contact
Stephen Parker	Base Coordinator	Tetra Tech	978-474-8400	Stephen.parker@tetratech.com	Base coordinator

Comments/Decisions: G. Lombardo provided email feedback requested at the September 16, 2009 NAVSTA RPM Meeting. Comments included the following:

- Address EPA's May 16, 2005 comments and Navy's June 15, 2005 RTCs in the SASE WP.
- Depth of fill? Did the test pits completed provide adequate information on the depth of fill throughout the site? Soil characterization should address the extent of fill material.
- Navy contends that elevated PAHs are caused by stormwater runoff from upgradient paved areas. The source is still a Navy source. The risk screening would still need to be completed for PAHs in soil.
- Navy contends elevated arsenic in soil (4.1 – 17.7 mg/kg) is consistent with background. Groundwater arsenic and lead concentrations were greater than MCLs, but this was unfiltered groundwater from the bottom of test pits. Need to complete adequate groundwater characterization.
- Surface water and sediment need to be sampled for potential contaminants.
- Both Human Health and Ecological Risk Screening Evaluations need to be included.

Action Items: Need to complete a risk screening and then support that the elevated levels in soil are a background condition.

Consensus Decisions: Feedback contained in the email was used to develop project-specific DQOs.

SAP Worksheet #9c -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Site 04, Coddington Cove Rubble Fill Area Projected Date(s) of Sampling: TBD Project Manager: Tom Campbell	Site Name: Site 04, Coddington Cove Rubble Fill Area Site Location: NAVSTA Newport, Rhode Island

Date of Session: November 16, 2009
Scoping Session Purpose: Email Correspondence from Tetra Tech

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Winoma Johnson	RPM	Navy	(757) 444-0825	winoma.johnson@navy.mil	Navy RPM
Paul Kulpa	State RPM	RIDEM	(401) 222-2797 ext. 7111	paul.kulpa@dem.ri.gov	State RPM
Ginny Lombardo	RPM	USEPA Region 1	617-918-1754	Lombardo.Ginny@epamail.epa.gov	Federal RPM
Cornelia Mueller	NAVSTA Newport Base Contact	Navy	401-841-7561	cornelia.mueller@navy.mil	Base Contact
Tom Campbell	PM	Tetra Tech	978-474-8400	Thomas.campbell@tetratech.com	Project Manager
Stephen Parker	Base Coordinator	Tetra Tech	978-474-8400	Stephen.parker@tetratech.com	Base coordinator

Comments/Decisions: Tetra Tech drafted an email containing the proposed DQO steps that will be incorporated into the Coddington Cove UFP SAP. EPA input contained in the October 22, 2009 email was incorporated. Input on the proposed DQO steps was requested from EPA, RIDEM, and Navy.

Consensus Decisions: Feedback in response to the email will be used to develop project-specific DQOs.

SAP Worksheet #9d -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Site 04, Coddington Cove Rubble Fill Area Projected Date(s) of Sampling: TBD Project Manager: Tom Campbell	Site Name: Site 04, Coddington Cove Rubble Fill Area Site Location: NAVSTA Newport, Rhode Island

Date of Session: December 10, 2009
Scoping Session Purpose: Email Correspondence from RIDEM

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Winoma Johnson	RPM	Navy	(757) 444-0825	winoma.johnson@navy.mil	Navy RPM
Paul Kulpa	State RPM	RIDEM	(401) 222-2797 ext. 7111	paul.kulpa@dem.ri.gov	State RPM
Ginny Lombardo	RPM	USEPA Region 1	617-918-1754	Lombardo.Ginny@epamail.epa.gov	Federal RPM
Cornelia Mueller	NAVSTA Newport Base Contact	Navy	401-841-7561	cornelia.mueller@navy.mil	Base Contact
Stephen Parker	Base Coordinator	Tetra Tech	978-474-8400	Stephen.parker@tetratech.com	Base coordinator
Tom Campbell	PM	Tetra Tech	978-474-8400	Thomas.campbell@tetratech.com	Project Manager

Comments/Decisions: P. Kulpa provided email feedback requested from a Tetra Tech email (November 16, 2009) that provided the proposed DQO steps and requested input from EPA, RIDEM, and Navy.

Comments included the following:

- There are some questions as to whether the test pits went to the base of the fill. As soil recovered from split-spoons in a rubble disposal area will probably be limited, and it will also be difficult to determine the depth of fill, it is recommended that test pits be excavated at the site (a larger excavator than that previously employed should also be considered).
- In regards to the PAH analysis, please confirm that it will be the full SVOC list.
- Please add TPH to the list of analytes (it was detected at the site above the criteria).
- VOCs were found in low concentrations in the soil and the groundwater. Please add VOCs to the list of analytes.
- The Navy has proposed installing overburden wells. If there are areas where the bedrock is shallow, the work plan should allow for the installation of the wells into the bedrock.
- Two chlorinated solvents were detected at low concentration. It is recommended that some of the wells be drilled into the bedrock and/or bedrock-overburden interface.

- In terms of sediment sampling, the work plan should include samples along the discharge route of the stream into the bay and at the discharge location into the bay.

- Prior to collecting sediment samples, holes 0 to 1.5 feet deep should be excavated and examined for field evidence of contamination. Sediment samples should be biased towards intervals which exhibit field evidence of contamination, if any.

  During the initial investigation, balls and other small toys were observed in the wetlands. Considering the proximity to Naval housing and the observations made at the site, the Navy should consider including the sediment samples in the human health risk assessment.

- It is recommended that flexibility be added into the work plan. As an illustration, if during the test pitting or soil boring program there is evidence of contamination, monitoring wells could be added and/or moved to these areas.

Action Item:

- Navy to clarify that full SVOC list will be analyzed for groundwater samples, and PAH list will be analyzed for in soil, sediment, and surface water samples.
- Navy recommends that TPH not be included the analysis list.
- Navy recommends adding VOCs to analyte list.

Consensus Decisions: Feedback contained in the email was used to develop project-specific DQOs.

SAP Worksheet #9e -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Site 04, Coddington Cove Rubble Fill Area Projected Date(s) of Sampling: TBD Project Manager: Tom Campbell	Site Name: Site 04, Coddington Cove Rubble Fill Area Site Location: NAVSTA Newport, Rhode Island

Date of Session: January 13, 2010
Scoping Session Purpose: Conference call with EPA to discuss EPA Comments on DQO Outline

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Winoma Johnson	RPM	Navy	(757) 444-0825	winoma.johnson@navy.mil	Navy RPM
Ginny Lombardo	RPM	USEPA Region 1	617-918-1754	Lombardo.Ginny@epamail.epa.gov	Federal RPM
Cornelia Mueller	NAVSTA Newport Base Contact	Navy	401-841-7561	cornelia.mueller@navy.mil	Base Contact
Tom Campbell	PM	Tetra Tech	978-474-8400	Thomas.campbell@tetratech.com	Project Manager

Comments/Decisions: Comments on the proposed DQO steps received from EPA by email on December 23, 2009.

Consensus Decisions: A conference call was conducted on January 13, 2010 to review EPA comments on the Coddington Cove Rubble Fill Area DQO outline.

- During the call, the TAL analyses proposed for groundwater samples were clarified to include VOCs, SVOCs, pesticides, and PCBs. In addition, TAL metals analysis will also be conducted for groundwater samples.

- The analyses proposed for soil samples were discussed. EPA's comment questioned why VOCs, SVOCs, and TPH analyses were not included. During the call it was agreed to add VOC analysis. Navy explained that full SVOC analysis was not proposed because a previous investigation detected PAHs and three phthalates in soil samples. It was agreed to limit the analyses to PAHs.

- EPA's request to include TPH analysis was discussed. Navy pointed out that DRO was detected in a previous investigation but only two samples exceeded the RIDEM residential criteria (500 mg/kg). A decision was held, pending internal EPA discussion on this topic.

The EPA comment requesting VOC analysis for sediment and surface water samples was discussed. It was agreed that VOC analysis would be included. In addition, sediment and surface water samples will be analyzed for PAHs, pesticides, PCBs, and metals.

SAP Worksheet #9f -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Site 04, Coddington Cove Rubble Fill Area Projected Date(s) of Sampling: TBD Project Manager: Tom Campbell	Site Name: Site 04, Coddington Cove Rubble Fill Area Site Location: NAVSTA Newport, Rhode Island

Date of Session: January 20, 2010
Scoping Session Purpose: NAVSTA Newport RPM Meeting

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Winoma Johnson (by phone)	RPM	Navy	(757) 444-0825	winoma.johnson@navy.mil	Navy RPM
Ginny Lombardo	RPM	USEPA Region 1	617-918-1754	Lombardo.Ginny@epamail.epa.gov	Federal RPM
Paul Kulpa	State RPM	RIDEM	(401) 222-2797 ext. 7111	paul.kulpa@dem.ri.gov	State RPM
Cornelia Mueller	NAVSTA Newport Base Contact	Navy	401-841-7561	cornelia.mueller@navy.mil	Base Contact
Tom Campbell	PM	Tetra Tech	978-474-8400	Thomas.campbell@tetratech.com	Project Manager

Comments/Decisions: The Coddington Cove Rubble Fill Area DQO outline was discussed at the January 20, 2010 NAVSTA Newport RPM meeting.

- EPA conveyed that potential petroleum-related contamination should be addressed along with CERCLA-related contaminants during preliminary sampling events. P. Kulpa conveyed that because DRO was detected above RIDEM residential criteria, TPH analysis would be requested by the State.

- Wetland sample locations were discussed. RIDEM requested a sediment sample in Coddington Cove. Navy responded that sediment sampling would be restricted to the site at this time. A sediment sample was proposed in the northernmost corner of the wetland where the onsite stream flows out of the site.

- P. Kulpa provided to Navy copies of historical aerial photographs that contained apparent evidence of filling activity in the southern portion of the onsite wetland.

Consensus Decisions: Incorporate decisions stated above into the Draft UFP SAP for Coddington Cove Rubble Fill Area.

NOTE: During comments and responses to comments on the draft version of this work plan, the Navy stated that TPH contamination at the site is understood from the PA sampling effort, and agreed that TPH would be analyzed only in the vicinity of the previous RIDEM residential criteria exceedances (i.e., TPH analysis will not occur at all locations during the SASE effort).

SAP Worksheet #10 – Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)

10.1 INTRODUCTION

Site 04, the Coddington Cove Rubble Fill (CCRF) Area (the site), is comprised of a former disposal/fill area and a wetland area. A Preliminary Assessment (PA) that included sampling was conducted at the site in May and June 2004. Sampling results indicated the presence of volatile organic compounds (VOCs), polycyclic aromatic hydrocarbons (PAHs), pesticides, polychlorinated biphenyls (PCBs), metals, and petroleum hydrocarbons in soil and groundwater seep samples. Based on these results, the PA concluded that the site should be carried forward to the Site Assessment Screening Evaluation (SASE) step in accordance with the Federal Facilities Agreement (FFA) for Naval Station (NAVSTA) Newport Installation Restoration (IR) Program sites (TtNUS, 2005).

10.2 SITE LOCATION, BACKGROUND, AND HISTORY

The CCRF Area is the location of a former disposal/fill area on an approximately 8 acre vacant lot located at NAVSTA Newport, near the southeastern portion of Coddington Cove, Narragansett Bay (Figure 10-1). The site is located between Coddington Highway to the southeast and the railroad right-of-way to the northwest (Figure 10-2).

The site consists of a now vegetated former fill area and a wetland area. The boundary between Newport and Middletown, Rhode Island crosses through the site in a southeast-northwest direction (Figure 10-2). Beyond the railroad right-of-way, which borders the site along its northwest boundary, are the Defense Highway and the southern portion of the On-Shore Derecktor Shipyard site (Site 19), which is bordered to the northwest by Coddington Cove, part of Narragansett Bay. To the north and east are a fenced, secure storage area and an open storage area. Located adjacent to the storage areas are a former railroad spur and NAVSTA Newport Building 47. A Navy housing development abuts the southwest boundary of the site and a chain link fence encloses the site along all boundaries (Figure 10-2).

Limited information is available regarding the historical land use of the CCRF Area site. According to a 1983 Initial Assessment Study (IAS), from 1978 to 1982 the site was a disposal area for inert rubble materials including concrete, asphalt, metal, slate, wood, brush, and a small amount of ash (Envirodyne, 1983). Reviewed records did not indicate additional land use practices.

The following text from the field sampling plan for the PA investigation at the CCRF Area describes the disposal areas, as interpreted from historical aerial photographs (Tetra Tech, 2004):

> "An aerial photograph from 1965 indicates two possible fill areas. A visible dirt drive extends from Coddington Highway to each suspected fill area. The fill areas are located adjacent to each other, in the center of the site and are approximately the same size. The areas encompass an estimated 900 square feet. Distinct mounds are visible in the northeast fill area along the northern edge….
>
> According to the aerial photograph from 1981…..the southern fill area identified in the 1965 photograph is no longer visible, as vegetation covers this area. The fill area to the north is still evident, though the extent has shifted further north. This fill area…has an estimated size of 500 square feet…"

In addition, two historical aerial photographs from 1972 and 1981 were obtained from the Rhode Island Water Resources Board. The photograph from 1972 indicates fill material in the central and northeastern portions of the site.

The aerial photograph from 1981 has a visible dirt road extending into the northern portion of the fill area. The southern portion of the fill area does not appear to be disturbed by any vehicular traffic.

10.3 SITE PHYSICAL CHARACTERISTICS

The following sections present available information regarding the topography, geology, and hydrogeology of the CCRF Area site.

10.3.1 Topography and Drainage

The former disposal area is located near the central portion of the site. At the edges of the disposal area, the site topography slopes downward into the wetland. This change in elevation is marked by a ridge which trends southwest-northeast through the central portion of the site, and then curves toward the southeast in the area of the Newport-Middletown border. The ridge, which denotes the approximate perimeter of the onsite disposal area, slopes approximately 8 to 10 feet downward toward the onsite wetland area. Onsite surface drainage in this area is expected to follow local topography (as viewed during a site reconnaissance) and flow radically toward the northwest, north, and northeast into the wetland area. However, along the railroad right-of-way which forms the northwestern boundary of the site, the ground surface slopes downward away from the railroad tracks and toward the southeast. Surface water runoff in this area is expected to flow southeast from the railroad right-of-way toward the site's wetland area.

The site is completely vegetated, with vegetation types ranging from grass to thorn-scrub in the central, southern, and western sections, to light, wooded vegetation in the northern section. The wetland area is thickly vegetated with a variety of wetland plant species.

10.3.2 Site Geology

Much of the overburden at the site consists of artificial fill materials, including brick, asphalt, plastic, ash, concrete, gravel, roots, and construction debris (sand, gravel, asphalt, concrete, and broken rock). The depth of fill material ranges from 8 to 10 feet below ground surface (bgs). Underlying the fill material is loam, silty sand, and gravel, in varying proportions.

The underlying bedrock is presumed to consist of slate or phyllite occurring at depths beginning at approximately 5 to 25 feet bgs. Test pits completed for a previous investigation did not encountered bedrock. The depth to the water table may be from 0 to 20 feet bgs in the wetland and fill areas, respectively. Test pits encountered groundwater from 8 to 10 feet bgs (TtNUS, 2005).

Not enough information is available to present an accurate geo cross-section for the CCRF Area site. The previous investigation was limited in scope. Once the SASE investigation is completed, this additional information will be included in the SASE Report.

10.3.3 Site Hydrogeology and Surface Water Hydrology

Overburden groundwater flow direction(s) at the site have not been determined, however, based on observed hydrologic conditions ((the presence of an on site wetland and stream and proximity and location of Coddington Cove are presumed to influence groundwater flow direction) at the site, flow is expected to be generally toward the northwest or north, into the site wetland area and/or the onsite stream.

The stream, which forms much of the north-northeastern boundary of the wetland area, enters the site through a storm drainage discharge located at the northeastern corner of the site, flowing from the direction of the Building 47 area. The stream flows approximately west and then north before exiting the site at the northernmost corner of the site. The stream then flows northeast along the railroad right-of-way before turning toward the northwest where it enters a culvert to Coddington Cove.

An underground sewer line (depth below ground surface unknown) transects the central portion of the site from southwest to northeast. A manhole associated with this sewer line is visible at the surface in the central portion of the site (Figure 10-2).

10.4 PREVIOUS INVESTIGATIONS

An IAS completed for Site 04 in March 1983 concluded that in the period from 1978 to 1982, the site was a disposal area for inert rubble materials, including concrete, asphalt, metal, slate, wood, brush, and a small amount of ash.

Field sampling was conducted in May and June 2004 as part of a Preliminary Assessment (PA). Samples collected included six surficial soil samples from the wetland area (interval collected was 0 to 1 foot bgs), 16 subsurface soil samples from test pits, (collected from depths below 1 foot bgs to a maximum depth of 10 feet bgs), and seven groundwater samples collected from groundwater that infiltrated into test pits, using the direct dip collection method. Soil samples were analyzed for VOCs, SVOCs, pesticides, PCBs, metals, gasoline-range organics (GRO), and diesel-range organics (DRO). Groundwater samples were analyzed for VOCs and metals. Sample locations from the PA are shown in Figure 10-2.

Analytical results from the PA investigation are presented below to identify the types and concentration ranges of chemical contaminants that were detected on site. In addition, this information is provided to identify specific areas on the CCRF Area site that were impacted by past disposal practices.

PA soil sample analytical results indicated the presence of VOCs at low concentrations. The following VOCs were detected in soils at the locations and maximum concentrations listed below (note that the qualifier "J" indicates the concentration is estimated): 1,4-dichlorobenzene (SS06, 4 J micrograms per kilogram [µg/kg]), benzene (SS01, 19 J µg/kg), chlorobenzene (SS06, 5 J µg/kg), ethylbenzene (12 J µg/kg), methylene chloride (SS01, 10 J µg/kg), and tetrachloroethene (TP02, 21 µg/kg). Acetone was also reported at 2,900 JEB µg/kg at TP10, however, this result was qualified as "JEB" due to possible equipment blank (EB) contamination. None of the concentrations detected exceeded the criteria selected in the PA report (Region 9 Preliminary Remediation Goals [PRGs] for Residential Soil).

Twenty SVOCs were detected in soil samples. Of these, five PAHs were detected above the selected criteria. All the maximum concentrations were detected at sample location SS04: benzo(a)anthracene (1,400 µg/kg), benzo(a)pyrene (1,700 µg/kg), benzo(b)fluoranthene (2,400 µg/kg), dibenzo(a,h)fluoranthene (360 µg/kg), and indeno(1,2,3-c,d)pyrene (1,200 µg/kg).

Three PCBs, Aroclor-1016, Aroclor-1254, and Aroclor-1260, were detected in soil samples. Aroclor-1260 was detected above the selected criterion in two intervals collected from test pit TP-07: 520 µg/kg at 2 to 4 feet bgs and 300 µg/kg at 6 to 8 feet bgs.

Nine pesticides were detected in soil samples. Dieldrin (in SS01 at 50 µg/kg) was the only pesticide detected above the selected criteria. Metals detected in soil samples above criteria included arsenic (in TP07 at 17.7 milligrams per kilogram [mg/kg]), iron (in TP06 at 39,800 mg/kg), and manganese (in SS06 at 2,310 mg/kg).

GROs were detected in two samples, TP03 (4,200 µg/kg) and TP05 (4,000 µg/kg). DROs were detected more frequently, in all but three soil samples. Concentrations above the Rhode Island Residential Direct Exposure Criterion (500 mg/kg) were detected in two samples, SS03 (670 mg/kg) and SS04 (730 mg/kg).

Samples collected by direct dip methods from groundwater that infiltrated into test pits indicated the presence of two VOCs at trace levels: tetrachloroethene (TP02, 2 J µg/L) and 1,1,2-trichloro-1,2,2-trifluoroethane (TP02, 11 µg/L). Two metals were detected at concentrations greater than the selected criteria, Maximum Contaminant Levels (MCLs): arsenic (TP01, 40.9 µg/L) and lead (TP03, 76 µg/L).

10.5 CONCEPTUAL SITE MODEL

A preliminary conceptual site model diagram is presented as Figure 10-3. The potential source areas of contamination include the areas where fill was previously placed, including portions of the wetland, and upgradient sources. The environmental media of interest are surface soil, subsurface soil, groundwater, surface water, and sediment. Contamination may be present in soil and sediment as a result of materials deposited in the former fill areas, including the wetland area. Surface water may be impacted by discharge of contaminated groundwater to the surface water, by fill materials that have been deposited in direct contact with surface water (in the wetland), and by leaching of contaminants from contaminated soils and sediments. Groundwater may be impacted by leaching of contaminants that may be present in fill materials and surrounding soils.

Possible contaminant migration routes include: groundwater to sediment and surface water, overburden soil/fill to groundwater, and soil/fill directly to surface water via overland runoff. Based on the PA sample results, contaminants are expected to include VOCs, PAHs, pesticides, PCBs, petroleum hydrocarbons, arsenic, iron, lead, and manganese.

Potential ecological receptors for contaminants in surface soil include terrestrial plants, terrestrial vertebrates and invertebrates, as well as reptiles and amphibians. Potential ecological receptors for contaminants in sediment include aquatic plants and aquatic vertebrates and invertebrates.

Potential human receptors for contaminants in surface soil include construction workers, hypothetical industrial workers, child and adult trespassers, child and adult recreational users, and hypothetical

residents. For contaminants in subsurface soil, construction workers and hypothetical residents are potential human receptors. For contaminants in groundwater, construction workers, hypothetical industrial workers, and hypothetical residents are potential human receptors.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

The data collected in this investigation will be used primarily by Tetra Tech and the Navy, in consultation with the other members of the Project Team. The Project Team includes Navy, U.S. Environmental Protection Agency (USEPA), and Rhode Island Department of Environmental Management (RIDEM) representatives. The following text describes the development of the data quality objectives (DQOs).

11.1 DEFINE PROBLEM

The site was a disposal area for rubble material from 1978 to 1982. An initial PA determined the presence of contaminants (trace VOCs, PAHs, pesticides, PCBs, petroleum hydrocarbons, and metals) in soil and groundwater seep samples (groundwater that infiltrated into test pits) collected from six wetland soil sample locations and 10 test pits at concentrations exceeding screening criteria (Region 9 Residential Soil PRGs and MCLs for soil and groundwater, respectively). The PA report concluded that further investigation was needed to address media not yet evaluated, or not adequately evaluated, including overburden groundwater, sediment, and surface water, to determine if a release under the Comprehensive Environmental Response Compensation and Liability Act (CERCLA) has occurred, and whether the site should undergo further evaluation in the IR program. In addition, the PA report recommended that a screening risk evaluation be conducted to determine whether contaminants may pose adverse risk to human or environmental receptors. The Project Team must investigate these additional media and determine the appropriate path forward based on the findings.

11.2 INPUTS TO THE DECISION

The following data are needed to resolve the problem described in Section 11,1:

VOC, SVOC, pesticide, PCB, and target analyte list (TAL) metal (total and dissolved) concentrations in groundwater. These analytical groups were selected based on the lack of reliable groundwater samples from the site and based on feedback from the Project Team. Analytical results will be compared to the following criteria:

- EPA Maximum Contaminant Levels (MCLs) (May 2009)

and if MCLs are not available,

- EPA Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites – Tap Water values (May 2010) (EPA, 2010). (Values for non-carcinogens have been adjusted by dividing by 10 to correspond to a Hazard Quotient (HQ) of 0.1).

VOC, PAH, pesticide, PCB, TAL metal, and TPH (GRO/DRO) concentrations in soil. These analytical groups were selected based on the analytical results from the PA investigation and feedback from the Project Team. The data generated during this investigation will be compared separately to human health and ecological risk criteria to evaluate risk to these two categories of receptors. The human health risk screening criteria for soil data comparison will be the EPA RSLs for Chemical Contaminants at Superfund Sites – Residential Soil values (May 2010) (EPA 2010). (Values for non-carcinogens have been adjusted by dividing by 10 to correspond to a HQ of 0.1). In addition, separate from the risk screening, soil analytical results will be compared to RIDEM Residential Direct Exposure Criteria (DEC) and Leachability Criteria, for comparison purposes. TPH analytical results will be compared to RIDEM Residential DEC for TPH.

The ecological risk screening criteria for soil data comparison will include:

- Canadian Council and Ministers of the Environment (CCME) Recommended Canadian Soil Quality Guidelines (CCME, 2006);
- The lower of the Oak Ridge National Laboratory (ORNL) plant and invertebrate benchmarks (Efroymson, et al., 1997a, 1997b);
- Ministry of Housing, Spatial Planning and Environment (MHSPE) Circular on target values and intervention values for soil remediation (MHSPE, 2000); and
- USEPA Ecological Soil Screening Levels (Eco-SSL) (EPA, 2009).

VOC, PAH, pesticide, PCB, and TAL metal (total and dissolved) concentrations in sediment and surface water. These analytical groups were selected based on the analytical results from the PA investigation and feedback from the Project Team. The ecological risk criteria for sediment are selected in the following order of preference, based on availability:

- Consensus-Based Threshold Effect Concentrations (TEC) (MacDonald, et al., 2000);
- Ontario Ministry of Environment and Energy (OMOE) Lowest Effect Level (LEL), (OMOE, 1993);
- USEPA Ecotox Thresholds, (EPA, 1996);
- National Oceanographic and Atmospheric Administration (NOAA) NOAA Screening Quick Reference Tables (Buchman, 2006); and
- Secondary Chronic Value (SCV) (Jones, et al., 1997).

The ecological risk criteria for surface water were selected in the following order of preference, based on availability:

- EPA National Recommended Water Quality Criteria (NRWQC) (EPA, 2006);
- Rhode Island Department of Environmental Management (RIDEM) Ambient Water Quality Criteria and Guidelines (RIDEM, amended May 2009);
- Secondary Chronic Values (SCV) (Suter and Tsao, 1996); and
- National Oceanographic and Atmospheric Administration (NOAA) NOAA Screening Quick Reference Tables (Buchman, 2006).

Chemical specific project action levels are presented on Worksheet #15 for each environmental medium.

11.3 BOUNDARIES OF THE STUDY

The media identified in the problem definition (groundwater, soil, sediment, and surface water) that must be investigated are those media that were or could have been contaminated by a chemical release, or to which released chemicals may have migrated.

The groundwater population of interest is the overburden aquifer underlying the former disposal area. To adequately investigate this population, the placement of overburden monitoring well screens across the water table will be determined by the field geologist using field observations and historic data. At selected locations where the PA reported VOC detections, well screens must be installed directly above the overburden/bedrock interface, where dense non-aqueous phase organic chemicals may accumulate. Groundwater at the southern perimeter must also be investigated to determine if potential up gradient sources are impacting the site. Total and dissolved metal groundwater fractions will be collected in case high turbidity levels are present. In addition, the dissolved fraction is more significant from an ecological risk perspective since this fraction is more bioavailable to aquatic organisms.

The soil population of interest is soil/fill within the footprint of the former location of the disposal area. The fill material is presumed to extend up to 10 feet below ground surface based on test pits completed during the PA.

Another population of interest is wetlands as indicated by the presence of hydric plants and soils and other physical feature unique to wetlands. The intent is to use these physical features to distinguish upland areas from wetlands.

The geographic area to be evaluated includes the former disposal area, consisting of upland areas, and the onsite wetland and fill areas. The site is enclosed by a chain-link fence and is bounded by

Coddington Cove Highway to the southeast, Navy housing to the southwest, railroad tracks right-of-way to the northwest, and Navy storage areas to the northeast.

There are no physical constraints on the timing for data collection; however, the project team wants to conclude the data collection at the earliest reasonable time.

11.4 ANALYTIC APPROACH

The chemical data will be used to make project decisions using the following decision rules:

1. In the former disposal area, if site target analyte concentrations in any groundwater or soil samples exceed the human health PALs, the Project Team will determine whether a removal action or a remedial investigation and a baseline risk assessment are required. The tendency will be to conduct a removal action if localized areas of relatively high contamination are identified that can be easily removed or an acute risk is anticipated from one or more areas at the site. If any target analyte concentration exceeds its PAL in any sample but a removal action is not warranted, the Project Team may recommend a Remedial Investigation (RI) considering potential risk management decisions. If all target analyte concentrations in all samples are less than their PALs a recommendation of no further investigation will be made by the Project Team.

   As part of the investigation of the former disposal area, the SASE investigators will use geological data and analytical data from test pits and soil borings to determine what contaminants at the site are related to fill materials disposed at the site. These determinations will be based on spatial concentration patterns that indicate connections with or overlap among chemicals and spatial locations. These data will provide further information on the composition and extent of fill to determine if the materials are regulated under CERCLA which may be useful for subsequent investigations.

2. If, based on a screening level ecological risk assessments (SLERA) of soil, sediment, and surface water analytical data from the 2004 PA and this SASE, there are unacceptable exceedances of target analyte concentrations relative to ecological risk screening levels, the Project Team may recommend an RI (to include a baseline ecological risk assessment) considering potential risk management decisions. The SLERA will consist of Steps 1, 2, and 3a of the eight steps in accordance with guidance documents (EPA, 1997, EPA, 1998, and DON, 1999). Figure 11-1 graphically depicts Steps 1, 2, and 3a from the Navy Ecological Risk Assessment Approach. If target analyte concentrations do not exceed PALs in any soil, sediment sample, and surface water sample a recommendation of no further investigation will be made by the Project Team.

11.5 PERFORMANCE CRITERIA

Measured concentrations of target analytes representing locations biased toward potential contamination will be compared to criteria. The Project Team will use the results of this investigation to determine whether the amount, type, and quality of data collected are sufficient to support the attainment of project objectives. This will involve an evaluation of contaminant concentrations, spatial contamination patterns, and an evaluation of uncertainty for contaminants that have criteria which are less than the MDLs to ensure that contaminants are likely to have been detected, if present. In addition, the Project Team will consider the possibility that some compounds can be naturally occurring, applied as intended (e.g. pesticides), or result from anthropogenic sources not related to a CERCLA release. A comparison to background values will also be conducted where applicable. Results of data validation (i.e., data qualifications) will be considered in these evaluations with an increased tendency to recommend additional investigation with decreased data quality. Data validation criteria are presented in Worksheets #35 and #36. The data usability assessment process is described in more detail in Worksheet #37.

11.6 SAMPLE DESIGN

The sampling strategy for this SASE Investigation is presented in Worksheet #17.

SAP Worksheet #12 -- Measurement Performance Criteria Table

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table – Field QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Equipment Rinsate Blanks	All Fractions	One per 20 field samples per matrix per sampling equipment[1].	Accuracy/Bias/ Contamination	No analytes ≥ ½ QL, except common lab contaminants, which must be < QL.	S&A
Trip Blanks	VOCs/GRO	One per cooler containing VOC samples.	Accuracy/Bias/ Contamination	No analytes ≥ ½ QL, except common lab contaminants, which must be < QL.	S&A
Matrix Spike (MS)	All Fractions	One per 20 field samples per matrix.	Accuracy/Bias	Within laboratory statistically derived percent recovery (%R) limits.	A
Matrix Spike Duplicate (MSD)	VOCs/GRO	One per 20 field samples per matrix.	Accuracy/Bias/ Precision	Within statistically derived %R limits; RPD of ≤30%[2] (aqueous).	A
Laboratory Duplicate	Metals	One per 20 field samples per matrix.	Precision	RPD of ≤30%[3] (aqueous).	A
Field Duplicate	All Fractions	One per 10 field samples collected.	Precision	Values > 5X QL: Relative Percent Difference (RPD) ≤30%[2, 3] (aqueous)	S
Cooler Temperature Indicator	All Fractions	One per cooler.	Representativeness	Temperature between 2 and 6 degrees Celsius (4 ± 2 °C).	S

1 – Equipment rinsate blanks will be collected if non-dedicated submersible pumps or other equipment are used.
2 – If duplicate values for non-metals are < 5x QL, the absolute difference should be < 2x QL.
3 – If duplicate values for metals are < 5x QL, the absolute difference should be < 4x QL.

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table
(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
PA Investigation of CCRF Area, Site 04	Results of Field Sampling Effort – May and June 2004, Coddington Cove Rubble Fill Area, SA-04 (Tetra Tech, 2005)	Originating organization: Tetra Tech Data types: Soil and groundwater seep analytical data (groundwater infiltrated in test pits) Data collection dates: May and June 2004	The data will be compared with new area data to evaluate whether the concentrations exceed human health and ecological risk criteria.	No overburden groundwater monitoring well data or surface water data are included

SAP Worksheet #14 -- Summary of Project Tasks
(UFP-QAPP Manual Section 2.8.1)

The following project tasks are summarized below:

- Field Tasks
- Analytical Tasks
- Data Management and Review
- Project Report

Standard operating procedures (SOPs) and field documentation forms are included in Appendix A and Appendix B, respectively. Project-specific procedures for applicable field tasks are included in Appendix C, including project-specific procedures for monitoring well development, water level measurement, and groundwater sampling in Appendix C-1; and procedures for soil sampling in Appendix C-2. The analytical laboratory specification is presented in Appendix D and the laboratory SOPs are presented in Appendix E.

14.1 FIELD TASKS

The project field tasks are summarized in this section.

14.1.1 Mobilization/Demobilization

Under this task, procurement of field equipment and supplies will be conducted. Personnel, subcontractors, equipment, and supplies will be mobilized to the field. This task also includes coordination with Dig-Safe and the NAVSTA Newport Environmental Office, a site walkover, and field orientation meetings to prepare for field work. Mobilization will be coordinated with the NAVSTA Newport Environmental Office a minimum of 1 week in advance of the initiation of field activities.

Tetra Tech will be responsible for obtaining clearance of all underground utilities at all sampling locations at least 72 hours prior to beginning onsite work. Tetra Tech will conduct a public and Navy Dig-Safe of the site. Tetra Tech will coordinate with local utility contractors and the NAVSTA Newport Environmental Office, marking drilling locations on the ground, visiting the site to meet with NAVSTA Newport Environmental Office utility marking crews, and making sample location adjustments if needed. Utility clearance will be performed in accordance with Tetra Tech SOP HS-1.0.

A field orientation meeting will be held prior to beginning the field program. The purpose of this meeting is to review the scope of the field investigation including site description, objectives, sampling locations, sampling methods, field QC samples, health and safety requirements, NAVSTA protocol and chain-of-

command. The field orientation meeting will be attended by the field staff, project manager, lead chemist, and health and safety officer. Field team members will review this SAP, applicable SOPs (Appendix A), and applicable Field Documentation Forms (Appendix B). All subcontractors working at the site will be provided with the site-specific health and safety plan, as well as their specifications and scopes.

Vehicles and portable sanitary facilities will be procured for the site. In addition, a lockable storage container will be procured for storage of drummed investigation-derived waste (IDW) at the site.

Areas to be investigated at the site will be cleared of brush, undergrowth, and small trees for safety reasons and for access. Brush will be chipped and allowed to remain onsite.

If necessary, and as directed by the Navy, Tetra Tech will keep the gated portion of the site locked during their tenure at the site.

Demobilization includes removing field equipment and supplies from the site, returning rented equipment, managing IDW, performing general site cleanup, organizing and finalizing field paperwork, and entering field records/data into the site database.

14.1.2 Test Pit Excavation and Sampling

Test pits shall be excavated at a minimum of four locations to characterize the fill material at the site. An estimated eight soil samples will be collected from test pits (two per test pit). Soil samples will be selected based on evidence of contamination as noted by visual staining, olfactory evidence of releases, or other indications. Samples will be taken directly from the excavator bucket and, with the exception of VOC samples, be placed into a decontaminated stainless steel bowl using pre-cleaned stainless steel or PVC Trowels. Samples will be evaluated for total VOCs via headspace analysis (in order to identify zones of contamination) and then containerized for laboratory analyses, as indicated in Worksheet #19, which includes analytical methods, sample containers, and preservation and holding time requirements for each analytical group. Worksheet #15c lists target analytes, the SASE project action limits (PALs), and the project quantitation limits (PQLs) that must be met by the laboratory. The soil samples will be collected according to SOP SA-1.3 and using the project-specific procedures described in Appendix C-1.

14.1.3 Drilling and Soil Sampling

Drilling activities will be conducted at the site in order to characterize the fill material and overburden geology, collect surface and subsurface soil samples for laboratory analysis, and install overburden groundwater monitoring wells. Soil borings will be advanced at a total of eight locations, all of which will be completed as overburden groundwater monitoring wells. The selected locations maximize the

potential for detecting groundwater contamination from on site sources and upgradient sources, if present.

Soil borings will be advanced from the ground surface to the top of bedrock using drive-and-wash drilling techniques, in accordance with the procedures described in SOP GH-1.3. Continuous split-barrel soil samples will be retrieved at 2-foot intervals from each of the eight boring locations.

The boreholes will be logged according to SOP GH-1.5. The soil will be visually classified using the Unified Soil Classification System (USCS) following American Society for Testing and Materials (ASTM) Method D-2488-98. Soil samples for laboratory analysis will be collected from the following depths: the 0 to 1 foot interval; the interval immediately above bedrock; and two intermediate intervals to be selected by the project geologist, as described in Appendix C-2. The soil samples will be collected according to SOP SA-1.3 and using the project-specific procedures described in Appendix C-1.

Jar-headspace screening for total VOCs will be conducted for each soil interval in order to indentify boring intervals containing contamination. It is presumed that fill material containing organic contamination was comingled with fill material containing inorganic contamination. This assumption is based on The headspace screening will be conducted using the procedures included in Appendix C-1. Following headspace screening, soils will be containerized for laboratory analyses as indicated in Worksheet #19, which includes analytical methods, sample containers, and preservation and holding time requirements for each analytical group (see Worksheets 19 and 20).

14.1.4 Monitoring Well Installation and Development

Overburden monitoring wells will be installed in each of the eight soil borings. The monitoring wells will be installed and developed according to SOP GH-2.8 using the project-specific procedures described in Appendix C-2.

Each well will be developed no sooner than 48 hours after well completion. In general, wells will be screened across intervals of interest as identified by the field geologist and the lead hydrogeologist. Wells screened in the shallow overburden will have 10-foot screens which will be installed to intersect the average estimated water table at each location, with the bottom of screen approximately 8 feet below the water table. Wells which are screened deeper in the overburden, below the water table, will also be constructed with 10-foot screens.

14.1.5 Groundwater Level Measurement and Groundwater Sampling

Groundwater samples will be collected from the eight newly-installed monitoring wells. A minimum of 1 week shall elapse after well development before collecting groundwater samples.

Prior to groundwater sampling, water levels will be measured in accordance with SOP GH-1.2 and using the project-specific procedures in Appendix C-2. Groundwater sample collection will be conducted according to USEPA's Low Stress (Low Flow) Purging and Sampling Procedure (EQASOP-GW 001, Rev. 3, January 19, 2010) and using the project-specific procedures presented in Appendix C-2. Procedures in Tetra Tech SOP SA-1.1 (Groundwater Sample Acquisition and Onsite Water Quality Testing) will be followed if a site-specific issue arises that is not covered by the above-referenced USEPA procedure. Rental bladder pumps and dedicated sample tubing will be used at each monitoring well to avoid potential cross-contamination between monitoring wells.

Water quality parameters will be measured during purging using a YSI Model 600 Series multi-parameter sonde or equivalent, coupled with a flow-through cell. Turbidity will be measured separately using a LaMotte Model 2020 or equivalent nephelometer.

Groundwater samples will be containerized for laboratory analyses as indicated in Worksheet #19, which includes analytical methods, sample containers, and preservation and holding time requirements for each analytical group.

14.1.6 Surface Water/Sediment Sampling

Seven collocated surface water and sediment samples will be collected in the wetland areas of the site and in the stream that bounds the site to the northeast (Figure 17-1).

Surface water and sediment samples will be containerized for laboratory analyses as indicated in Worksheet #19, which includes analytical methods, sample containers, and preservation and holding time requirements for each analytical group. Both surface water and sediment samples and associated quality control (QC) samples will be analyzed for VOCs, PAHs, pesticides, PCBs, and TAL metals. Worksheets #15b and #15d list target analytes, the SASE PALs, and the PQLs that must be met by the laboratory, for surface water and sediment samples, respectively. Additional information on the sediment sampling procedure and surface water sampling procedure is provided below.

Sediment Sampling Procedure: Sediment samples will be collected in accordance with SOP SA-1.2 (Appendix A) from a depth of 0 to 6 inches from the sediment surface, using a decontaminated stainless steel auger. Obvious signs of contamination observed by the sampling team will be preferentially

sampled. The sediment samples will be collected in accordance with SOP SA-1.2 and using the following procedure:

1. Record all required data for each sample on the sample log sheet. Include the sampling equipment, sampling personnel, date, time, depth of sample, and sample analyses. The sediment will be visually classified using the USCS Method.

2. Label appropriate sample containers with the sample location number, date, time, and analysis.

3. Carefully remove vegetation, roots, twigs, litter, etc., to expose the sediment surface area.

4. Using a decontaminated, stainless-steel hand auger, collect a sediment core representative of the required sample interval.

5. While sediment is still in the auger material will be collected using a cut syringe or equivalent and transferred into the appropriate VOC vials using the procedures described in SOP SA-1.2.

6. Transfer the sediment from the hand auger to a decontaminated stainless-steel bowl using only dedicated or decontaminated stainless steel spoons or trowel, and homogenize the sample.

7. Remove any large particles such as gravel or artificial fill too large to be sent for analysis. Note the removal of material on the sample log sheet.

8. Fill the appropriate sample containers.

9. For field duplicate samples, after homogenization, fill one set of sample containers for the original sample and fill another set for the field duplicate sample.

10. Ensure that the samples are properly labeled, maintained in coolers with ice, and that the chain-of-custody procedures described in Worksheet #27 are followed. Package and prepare for pick-up or ship the sample coolers to the appropriate laboratory for overnight delivery.

11. Decontaminate the sampling equipment before reuse.

Surface Water Sampling Procedure: Collocated surface water samples will be collected at each sediment sample location. Each surface water sample will be collected by direct dipping the sample containers. Stream samples will be collected from downstream to upstream locations. All surface water samples will

be collected following Tetra Tech SOP SA-1.2. After collection of surface water samples, the pH, temperature, and dissolved oxygen of surface water will be measured in-situ and recorded.

14.1.7 Land Surveying

After sample collection, the horizontal coordinates and ground surface elevations of all soil borings/monitoring wells, test pits, sediment/surface water sample locations, and other pertinent features will be determined by a Rhode Island registered land surveyor. The coordinates of the features will be incorporated into the NAVSTA Newport Geographic Information System (GIS) database and used for site mapping.

At all monitoring wells, additional elevation measurements will be taken at the top of the inner PVC casing and the top of the protective steel casing. All vertical measurements will be surveyed in United States Geological Survey (USGS) National Geodetic Vertical Datum of 1929 (NGVD 1929) coordinates, in feet. All horizontal measurements will be in Rhode Island State Plane coordinates, using 1983 North American Datum (NAD 1983), in feet.

Horizontal locations will be measured to the nearest 0.1 foot NAD 1983. Elevations of well risers and protective casings will be measured to the nearest 0.01 foot NGVD 1929. Vertical and horizontal control will be brought to the site using local benchmark information and temporary benchmarks will be established as needed.

The subcontractor deliverable will include a base map of the site, showing existing permanent features as well as sample stations (borings/wells, test pits, sediment/surface water sample locations, and other features of interest).

14.1.8 Quality Control Tasks

Field QC samples will be collected as part of the investigation, including field duplicates, trip blanks, and equipment rinsate blanks for the fixed laboratory analyses.

Also, samples will be assigned for the laboratory to perform MS/MSD analysis (for groundwater samples and soil samples). Worksheet #20 presents the field QC sample summary.

14.1.9 Calibration Procedures

Field instrument calibration procedures are presented in Worksheet #22.

14.1.10 Decontamination

The decontamination procedures presented in SOP SA-7.1 will be followed. Non-disposable equipment that comes in contact with the sample medium will be decontaminated to prevent cross-contamination between sampling points. This includes equipment such as augers, stainless steel bowls, scoops, as well as heavy equipment. All heavy equipment, including the drilling rig and downhole equipment used during property investigation activities will be decontaminated prior to beginning work and between all boreholes using a high-pressure steam wash with potable water. Water level indicators will be sprayed with de-ionized water and wiped with clean paper towels in between use in each monitoring well. Personnel decontamination is discussed in the Health and Safety Plan (HASP). The HASP will be prepared as a separate document prior to field activities.

14.1.11 Investigation-Derived Waste

IDW includes decontamination fluid, used personal protective equipment (PPE), disposable sampling equipment, excess drill cuttings, and excess soil samples. IDW will be managed in accordance with SOP SA-7.1, as follows:

- Soil cuttings and excess soil samples, decontamination rinse water, and water generated during purging of the wells during development and groundwater sampling will be collected and transferred for staging in 55-gallon drums or bulk containers. Separate drums will be used for soil and water. The containers with purge and decontamination water will be staged at the site inside a container box. Pending water and soil waste characterization, the drums containing wastewater and soil will be transported and disposed by a licensed contractor at an off-site facility licensed to accept the waste. IDW characterization, transport, and disposal will be performed after all IDW collection has been completed and properly containerized.

- Disposable personal protective equipment and disposable sampling equipment/materials (spent tubing, etc.) will be disposed of as general refuse at the approved disposal location on NAVSTA Newport property.

14.2 ANALYTICAL TASKS

Chemical analysis of groundwater, soil, sediment, and surface water samples will be performed by a subcontracted laboratory, Trimatrix Laboratories. Tetra Tech will perform the field screening using a PID and jar headspace procedures.

Trimatrix Laboratories will perform chemical analyses in accordance with the methods identified in Worksheet #19 and the requirements of the analytical specification for laboratory services developed by Tetra Tech for this work (Appendix D). Trimatrix Laboratories will perform the chemical analyses following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the methods listed in Worksheet #19. Copies of the Laboratory SOPs are included in Appendix E.

14.3 DATA MANAGEMENT AND REVIEW

Data management and review tasks are described in worksheets in this SAP as identified below.

Project documentation and records

- Field sample collection and field measurement records are described in Worksheets #27 and #29.
- Laboratory data package deliverables are described in the analytical specifications.
- Data assessment documents and records are listed in Worksheet #29.

Data recording formats – These formats are described in Worksheet #27.

Data handling, management, tracking and control – These processes are described in Worksheet #29.

Data Review

- Data verification processes are described in Worksheet #34.
- Data validation processes are described in Worksheets #35 and #36.
- Usability assessment processes are described in Worksheet #37.

SASE Report

Tetra Tech will first prepare an Internal Draft SASE Report for Navy review. The Internal Draft Report will include the following sections:

- Executive Summary – a very brief description of the work conducted and the findings.

- Introduction and Background – includes a description of the history of operations and activities at the site and a summary of the previous investigation.

- Description of Field Investigations – includes a summary of the work planned and described in this QAPP and any field modification as documented by the field geologist and engineers. This section will include maps showing the sampling station locations and well installations and tables summarizing the data collected.

- Physical Features of the Site – this section will describe the geological, hydrogeological, and topographical features of the site; the wetland portions of the site (surface water and sediment conditions); and the ecological setting of the site.

- Nature of Contamination – includes the contamination detected in each environmental medium sampled in relation to the conceptual model of the site. Areas confirmed and likely to have been impacted by contaminants will be described and mapped. Possibility for contaminants to be present beyond the sample areas will be discussed. Detected concentration of contaminants will be presented in tables for each medium sampled and on maps.

- Contaminant Fate and Transport – includes a description of the contaminants detected and their behavior in the soil, sediment, surface water, or groundwater media, particularly with emphasis on the future migration of these contaminants either through the food chain or other medium.

- Streamlined Risk Evaluation – the initial stage of risk evaluations, limited to comparison of contaminant concentrations found in the samples to human health risk criteria and ecological effects criteria. The findings of the assessment will identify if contaminants are present at the site at levels that may be of concern to human health or the environment.

- Refined Conceptual Site Model – the conceptual site model will be refined according to the data collected, the geological conditions encountered, and other physical characteristics of the site.

- Summary and Conclusions – this section will summarize the findings; describe the need for additional ecological assessments, if any; identify additional data collection needs, if any; and conclude if a Remedial Investigation and Baseline Risk Assessment are required, based on the findings.

The Internal Draft Report will be revised to address Navy comments and a Draft SASE Report will be prepared and distributed to RIDEM, EPA, and Navy. Electronic copies will be provided to Gannett Fleming and the NAVSTA RAB.

After receipt of review comments from EPA and RIDEM, Tetra Tech will prepare one set of response to comments (RTCs). A meeting with Navy and regulators may be held to resolve outstanding questions/issues on the regulatory comments prior to completion of a Draft Final SASE Report. The Draft Final SASE Report will be prepared and distributed to RIDEM, EPA, and Navy. Electronic copies will be provided to Gannett Fleming and the NAVSTA RAB.

After receipt of review comments on the Draft Final SASE Report from EPA and RIDEM, Tetra Tech will prepare one set of RTCs. Any outstanding questions/issues on the SASE Report will be resolved via conference call prior to completion of the Final SASE Report, which will be prepared and distributed to RIDEM, EPA, and Navy. Electronic copies will be provided to Gannett Fleming and the NAVSTA Document repositories.

SAP Worksheet #15a -- Groundwater Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Groundwater Samples

Groundwater Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
VOCs (ug/L)								
1,1,1-Trichloroethane	71-55-6	8260B	200	MCL	66.7	1.0	0.50	0.19
1,1,2,2-Tetrachloroethane	**79-34-5**	**8260B**	**0.067**	**RSL**	**0.02**	**1.0**	**0.25**	**0.070**
1,1,2-Trichloroethane	79-00-5	8260B	5	MCL	1.7	1.0	0.50	0.18
1,1-Dichloroethane	75-34-3	8260B	2.4	RSL	0.8	1.0	0.50	0.13
1,1-Dichloroethene	75-35-4	8260B	7	MCL	2.3	1.0	0.50	0.13
1,2,4-Trichlorobenzene	120-82-1	8260B	70	MCL	23.3	1.0	0.50	0.16
1,2-Dibromoethane (EDB)	**106-93-4**	**8260B**	**0.05**	**MCL**	**0.02**	**1.0**	**0.50**	**0.22**
1,2-Dibromo-3-chloropropane (DBCP)	**96-12-8**	**8260B**	**0.2**	**MCL**	**0.1**	**1.0**	**1.0**	**0.40**
1,2-Dichlorobenzene	95-50-1	8260B	600	MCL	200.0	1.0	0.50	0.12
1,2-Dichloroethane	107-06-2	8260B	5	MCL	1.7	1.0	0.50	0.13
1,2-Dichloropropane	78-87-5	8260B	5	MCL	1.7	1.0	0.50	0.15
1,3-Dichlorobenzene	541-73-1	8260B	--	--	--	1.0	0.50	0.13
1,4-Dichlorobenzene	106-46-7	8260B	75	MCL	25.0	1.0	0.50	0.12
2-Butanone	78-93-3	8260B	710	RSL	237	5.0	1.0	0.55
2-Hexanone	**591-78-6**	**8260B**	**4.7**	**RSL**	**1.6**	**5.0**	**1.0**	**0.29**
4-Methyl-2-pentanone	108-10-1	8260B	200	RSL	66.7	5.0	0.50	0.28
Acetone	67-64-1	8260B	2200	RSL	733.3	10.0	5.0	2.1
Benzene	71-43-2	8260B	5	MCL	1.7	1.0	0.50	0.18
Bromodichloromethane	75-27-4	8260B	80	MCL	26.7	1.0	0.50	0.13
Bromoform	75-25-2	8260B	80	MCL	26.7	1.0	0.50	0.15

Groundwater Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Bromomethane	74-83-9	8260B	--	--	--	--[4]	--[4]	--[4]
Carbon disulfide	75-15-0	8260B	100	RSL	33.3	5.0	0.50	0.18
Carbon tetrachloride	56-23-5	8260B	5	MCL	1.7	1.0	0.50	0.13
Chlorobenzene	108-90-7	8260B	100	MCL	33.3	1.0	0.50	0.19
Chloroethane	75-00-3	8260B	2100	RSL	700.0	1.0	0.50	0.15
Chloroform	67-66-3	8260B	80	MCL	26.7	1.0	0.50	0.17
Chloromethane	74-87-3	8260B	19	RSL	6.3	1.0	0.50	0.16
cis-1,2-Dichloroethene	156-59-2	8260B	70	MCL	23.3	1.0	0.50	0.23
cis-1,3-Dichloropropene	**10061-01-5**	**8260B**	**0.43**	**RSL**	**0.1**	**1.0**	**0.50**	**0.25**
Cyclohexane	110-82-7	8260B	1300	RSL	433.3	5.0	0.50	0.25
Dibromochloromethane	124-48-1	8260B	80	MCL	26.7	1.0	0.50	0.14
Dichlorodifluoromethane	75-71-8	8260B	39	RSL	13.0	1.0	0.50	0.21
Ethylbenzene	100-41-4	8260B	700	MCL	233.3	1.0	0.50	0.14
Isopropylbenzene	98-82-8	8260B	68	RSL	22.7	1.0	0.50	0.15
Methyl acetate	79-20-9	8260B	3700	RSL	1233	5.0	0.50	0.30
Methylcyclohexane	108-87-2	8260B	--	--	--	5.0	0.50	0.20
Methylene chloride	75-09-2	8260B	5	MCL	1.7	1.0	0.50	0.26
Methyl-tert-butyl ether	1634-04-4	8260B	12	RSL	4	1.0	0.50	0.28
Styrene	100-42-5	8260B	100	MCL	33.3	1.0	0.50	0.11
Tetrachloroethene	127-18-4	8260B	5	MCL	1.7	1.0	0.50	0.16
Toluene	108-88-3	8260B	1000	MCL	333.3	1.0	0.50	0.16
trans-1,2-Dichloroethene	156-60-5	8260B	100	MCL	33.3	1.0	0.50	0.20
trans-1,3-Dichloropropene	10061-02-6	8260B	0.43	RSL	0.1	1.0	0.50	0.23
Trichloroethene	79-01-6	8260B	5	MCL	1.7	1.0	0.25	0.092
Trichlorofluoromethane	75-69-4	8260B	130	RSL	43.3	1.0	0.50	0.20

Groundwater Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Vinyl chloride	75-01-4	8260B	2	MCL	0.7	1.0	0.25	0.10
Xylenes (total)	1330-20-7	8260B	10000	MCL	3333	3.0	1.0	0.42
SVOCs (ug/L)								
1,1'-Biphenyl	92-52-4	8270C	180	RSL	60.0	0.5	0.05	0.10
1,4-Dioxane	123-91-1	8270C SIM	6.1	RSL	3	1.0	1.0	0.37
2,2'-Oxybis(1-chloropropane)	108-60-1	8270C	0.32	RSL	0.11	0.50	0.10	0.059
2,4,5-Trichlorophenol	95-95-4	8270C	370	RSL	123.3	0.50	0.10	0.099
2,4,6-Trichlorophenol	88-06-2	8270C	6.1	RSL	2.0	0.50	0.10	0.059
2,4-Dichlorophenol	120-83-2	8270C	11	RSL	3.7	0.50	0.10	0.056
2,4-Dimethylphenol	105-67-9	8270C	73	RSL	24.3	1.0	0.50	0.24
2,4-Dinitrophenol	51-28-5	8270C	7.3	RSL	2.4	5.0	1.0	2.2
2,4-Dinitrotoluene	121-14-2	8270C	0.22	RSL	0.1	1.0	0.25	0.096
2,6-Dinitrotoluene	606-20-2	8270C	3.7	RSL	1.2	0.50	0.25	0.13
2-Chloronaphthalene	91-58-7	8270C	290	RSL	96.7	0.50	0.10	0.029
2-Chlorophenol	95-57-8	8270C	18	RSL	6.0	0.50	0.10	0.080
2-Methylphenol (o-cresol)	95-48-7	8270C	180	RSL	60.0	0.50	0.05	0.044
2-Nitroaniline	88-74-4	8270C	37	RSL	12.3	1.0	0.25	0.16
2-Nitrophenol	88-75-5	8270C	--	--	--	0.50	0.10	0.071
3,3'-Dichlorobenzidine	91-94-1	8270C	0.15	RSL	0.1	1.0	1.0	0.64
3-Nitroaniline	99-09-2	8270C	--	--	--	1.0	0.50	0.050
4,6-Dinitro-2-methylphenol	534-52-1	8270C	0.29--	RSL	0.1	0.50	0.25	0.17
4-Bromophenyl phenyl ether	101-55-3	8270C	--	--	--	0.50	0.05	0.036
4-Chloro-3-methylphenol	59-50-7	8270C	370	RSL	123	0.50	0.10	0.031
4-Chloroaniline	**106-47-8**	**8270C**	**0.34**	**RSL**	**0.1**	**1.0**	**0.25**	**0.15**
4-Chlorophenyl phenyl	7005-72-3	8270C	--	--	--	0.50	0.05	0.031

Groundwater Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
ether								
4-Methylphenol (p-cresol)	106-44-5	8270C	18	RSL	6.0	0.50	0.25	0.18
4-Nitroaniline	100-01-6	8270C	3.4	RSL	1.1	1.0	0.50	0.070
4-Nitrophenol	824-78-2	8270C	--	--	--	10	0.25	0.19
Acetophenone	98-86-2	8270C	370	RSL	123.3	0.50	0.10	0.068
Atrazine	1912-24-9	8270C	3	MCL	1.0	0.50	0.10	0.051
Benzaldehyde	100-52-7	8270C	370	RSL	123.3	0.50	0.10	0.22
Bis(2-chloroethoxy)methane	111-91-1	8270C	11	RSL	3.7	0.50	0.05	0.035
Bis(2-chloroethyl)ether	**111-44-4**	**8270C**	**0.012**	**RSL**	**0.004**	**0.50**	**0.05**	**0.035**
Bis(2-ethylhexyl)phthalate	117-81-7	8270C	6	MCL	2.0	2.0	0.25	0.24
Butyl benzyl phthalate	85-68-7	8270C	35	RSL	11.7	1.0	0.10	0.058
Caprolactam	105-60-2	8270C	1800	RSL	600.0	1.0	1.0	0.21
Carbazole	86-74-8	8270C	--	--	--	0.50	0.25	0.047
Dibenzofuran	132-64-9	8270C	3.7	RSL	1.2	0.50	0.05	0.039
Diethyl phthalate	84-66-2	8270C	2900	RSL	966.7	0.50	0.05	0.043
Dimethyl phthalate	131-11-3	8270C	--	--	--	0.50	0.05	0.045
Di-n-butyl phthalate	84-74-2	8270C	370	RSL	123	2.0	0.50	0.27
Di-n-octyl phthalate	117-84-0	8270C	--	--	--	1.0	0.10	0.064
Hexachlorobenzene	118-74-1	8270C SIM	1	MCL	0.3	0.50	0.10	0.062
Hexachlorobutadiene	87-68-3	8270C	0.86	RSL	0.3	0.50	0.10	0.057
Hexachlorocyclopentadiene	77-47-4	8270C	50	MCL	16.7	0.50	0.10	0.057
Hexachloroethane	67-72-1	8270C	4.8	RSL	1.6	0.50	0.10	0.035
Isophorone	78-59-1	8270C	71	RSL	23.7	0.50	0.10	0.056
Nitrobenzene	**98-95-3**	**8270C**	**0.12**	**RSL**	**0.04**	**0.50**	**0.10**	**0.076**
N-Nitrosodi-n-propylamine	621-64-7	8270C	0.0096	RSL	0.003	0.50	0.10	0.044
N-Nitrosodiphenylamine	86-30-6	8270C	14	RSL	4.7	0.50	0.05	0.042

Groundwater Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Pentachlorophenol	87-86-5	8270C	1	MCL	0.3	1.0	0.1	--[4]
Phenol	108-95-2	8270C	1100	RSL	366.7	0.50	0.05	0.49
PAHs (ug/L)								
2-Methylnaphthalene	91-57-6	8270C SIM	15	RSL	5	0.020	0.0040	0.0040
Acenaphthene	83-32-9	8270C SIM	220	RSL	73.3	0.020	0.0040	0.0040
Acenaphthylene	208-96-8	8270C SIM	220	RSL	73.3	0.020	0.0040	0.0040
Anthracene	120-12-7	8270C SIM	1100	RSL	366.7	0.020	0.0040	0.0040
Benzo(a)anthracene	56-55-3	8270C SIM	0.029	RSL	0.01	0.020	0.0040	0.0040
Benzo(a)pyrene	50-32-8	8270C SIM	0.2	MCL	0.1	0.020	0.0040	0.0040
Benzo(b)fluoranthene	205-99-2	8270C SIM	0.029	RSL	0.01	0.020	0.0040	0.0040
Benzo(g,h,i)perylene	191-24-2	8270C SIM	110	RSL	36.7	0.020	0.0040	0.0040
Benzo(k)fluoranthene	207-08-9	8270C SIM	0.29	RSL	0.1	0.020	0.0040	0.0040
Chrysene	218-01-9	8270C SIM	2.9	RSL	1.0	0.020	0.0040	0.0040
Dibenzo(a,h)anthracene	**53-70-3**	**8270C SIM**	**0.0029**	**RSL**	**0.001**	**0.020**	**0.0040**	**0.0040**
Fluoranthene	206-44-0	8270C SIM	150	RSL	50.0	0.020	0.0040	0.0040
Fluorene	86-73-7	8270C SIM	150	RSL	50.0	0.020	0.0040	0.0040
Indeno(1,2,3-cd)pyrene	193-39-5	8270C SIM	0.029	RSL	0.01	--[4]	--[4]	--[4]
Naphthalene	91-20-3	8270C SIM	0.14	RSL	0.05	0.020	0.0040	0.0040

Groundwater Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Phenanthrene	85-01-8	8270C SIM	110	RSL	36.7	0.020	0.0040	0.0040
Pyrene	129-00-0	8270C SIM	110	RSL	36.7	0.020	0.0040	0.0040
Pesticides (ug/L)								
4,4'-DDE	72-55-9	8081A	0.2	RSL	0.1	0.010	0.0008	0.00072
4,4'-DDD	72-54-8	8081A	0.28	RSL	0.1	0.010	0.0008	0.00058
4,4'-DDT	50-29-3	8081A	0.2	RSL	0.1	0.010	0.0008	0.00065
Aldrin	**309-00-2**	**8081A**	**0.004**	**RSL**	**0.001**	**0.010**	**0.0008**	**0.00080**
alpha-BHC	319-84-6	8081A	0.011	RSL	0.004	0.010	0.0008	0.00063
alpha-Chlordane	5103-71-9	8081A	2	MCL	0.7	0.010	0.0008	0.00052
beta-BHC	319-85-7	8081A	0.037	RSL	0.01	0.010	0.0008	0.00052
delta-BHC	319-86-8	8081A	0.2	RSL	0.1	0.010	0.0008	0.0011
Dieldrin	**60-57-1**	**8081A**	**0.0042**	**RSL**	**0.001**	**0.010**	**0.0008**	**0.00048**
Endosulfan I	959-98-8	8081A	22	RSL	7.3	0.010	0.0008	0.00046
Endosulfan II	33213-65-9	8081A	22	RSL	7.3	0.010	0.0008	0.00045
Endosulfan sulfate	1031-07-8	8081A	22	RSL	7.3	0.010	0.0008	0.00076
Endrin	72-20-8	8081A	2	MCL	0.7	0.010	0.0008	0.0062
Endrin aldehyde	7421-93-4	8081A	2	MCL	0.7	0.010	0.008	0.0039
Endrin ketone	53494-70-5	8081A	2	MCL	0.7	0.010	0.008	0.00093
gamma-BHC (Lindane)	58-89-9	8081A	0.2	MCL	0.1	0.010	0.0008	0.00064
gamma-Chlordane	5103-74-2	8081A	2	MCL	0.7	0.010	0.0008	0.00046
Heptachlor	76-44-8	8081A	0.4	MCL	0.1	0.010	0.0008	0.00043
Heptachlor epoxide	1024-57-3	8081A	0.2	MCL	0.1	0.010	0.0008	0.00056
Methoxychlor	72-43-5	8081A	40	MCL	13.3	0.010	0.008	0.0014
Toxaphene	8001-35-2	8081A	3	MCL	1.0	0.10	0.033	0.0087
PCBs (ug/L)								

Groundwater Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Aroclor-1016	12674-11-2	8082	0.5	MCL	0.2	0.10	0.09	0.025
Aroclor-1221	11104-28-2	8082	0.5	MCL	0.2	0.10	0.12	0.029
Aroclor-1232	11141-16-5	8082	0.5	MCL	0.2	0.10	0.08	0.032
Aroclor-1242	53469-21-9	8082	0.5	MCL	0.2	0.10	0.10	0.040
Aroclor-1248	12672-29-6	8082	0.5	MCL	0.2	0.10	0.10	0.030
Aroclor-1254	11097-69-1	8082	0.5	MCL	0.2	0.10	0.08	0.033
Aroclor-1260	11096-82-5	8082	0.5	MCL	0.2	0.10	0.09	0.026
Total Polychlorinated Biphenyls	1336-36-3	--	0.5	MCL	0.2	0.10	0.10	0.040
Metals (Total and Dissolved) (ug/L)								
Aluminum	7429-90-5	6010C	3700	RSL	1233.3	50	5.0	23
Antimony	7440-36-0	6020A	6	MCL	2.0	2.0	1.0	0.36
Arsenic	7440-38-2	6020A	10	MCL	3.3	5.0	1.0	0.38
Barium	7440-39-3	6020A	2000	MCL	666.7	2.0	1.0	0.59
Beryllium	7440-41-7	6020A	4	MCL	1.3	1.0	1.0	0.30
Cadmium	7440-43-9	6020A	5	MCL	1.7	0.20	0.20	0.055
Calcium	7440-70-2	6010C	--	--	--	500	100	67
Chromium	7440-47-3	6020A	100	MCL	33.3	1.0	1.0	0.46
Cobalt	7440-48-4	6020A	1.1	RSL	0.4	1.0	0.10	0.042
Copper	7440-50-8	6020A	1300	MCL	433.3	1.0	1.0	0.71
Iron	7439-89-6	6010C	2600	RSL	866.7	20	10	4.2
Lead	7439-92-1	6020A	15	MCL	5.0	1.0	0.50	0.22

Groundwater Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Magnesium	7439-95-4	6010C	--	--	--	500	100	48
Manganese	7439-96-5	6020A	88	RSL	29.3	2.0	1.0	0.42
Mercury	7439-97-6	7470A	2	RIDEM GA Aquifer	0.7	0.20	0.10	0.061
Nickel	7440-02-0	6020A	100	RIDEM GA Aquifer	33.3	1.0	1.0	0.37
Potassium	7440-09-7	6010C	--	--	--	500	100	82
Selenium	7782-49-2	6020A	50	MCL	16.7	5.0	1.0	0.82
Silver	7440-22-4	6020A	18	RSL	6.0	0.50	0.20	0.061
Sodium	7440-23-5	6010C	--	--	--	500	250	140
Thallium	7440-28-0	6020A	2	MCL	0.7	0.20	0.20	0.056
Vanadium	7440-62-2	6020A	18	RSL	6	5.0	1.0	0.28
Zinc	7440-66-6	6020A	1100	RSL	366.7	10	4.0	2.9

-- = Not available or not applicable.

1. All methods follow SW-846.
2. MCL = U.S. EPA Maximum Contaminant Levels for drinking water
   RSL = EPA RSLs for Chemical Contaminants at Superfund Sites, Tapwater Values (EPA 2009). One-tenth RSLs are displayed for noncancer RSLs to represent a target hazard quotient of 0.1.
3. Laboratory-specific LOQ, LODs, and MDLs are limits that Trimatrix Laboratory achieves when performing the specific analytical method.
4. Laboratory-specific LOQ, LODs, and MDLs limits for bromomethane, indeno(1,2,3-cd)pyrene, and pentachlorophenol have not been provided by Trimatrix Laboratory but will be added when available.

Bold rows indicate that the PAL is less than the corresponding QL but greater than the corresponding MDL.
Bold and shaded rows indicate that the PAL is less than the corresponding QL and the MDL.

SAP Worksheet #15b – Surface Water Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water Samples

Surface Water Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
VOCs (ug/L)								
1,1,1-Trichloroethane	71-55-6	8260B	11	SCV	3.7	1.0	0.50	0.19
1,1,2,2-Tetrachloroethane	79-34-5	8260B	10	RIDEM	3.3	1.0	0.25	0.070
1,1,2-Trichloroethane	79-00-5	8260B	20	RIDEM	6.7	1.0	0.50	0.18
1,1-Dichloroethane	75-34-3	8260B	47	SCV	15.7	1.0	0.50	0.13
1,1-Dichloroethene	75-35-4	8260B	13	RIDEM	4.3	1.0	0.50	0.13
1,2,4-Trichlorobenzene	87-61-6	8260B	1.7	RIDEM	0.6	1.0	0.50	0.16
1,2-Dibromoethane (EDB)	106-93-4	8260B	--	--	--	1.0	0.50	0.22
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	8260B	--	--	--	1.0	1.0	0.40
1,2-Dichlorobenzene	95-50-1	8260B	1.8	RIDEM	0.6	1.0	0.50	0.12
1,2-Dichloroethane	107-06-2	8260B	131	RIDEM	43.7	1.0	0.50	0.13
1,2-Dichloropropane	78-87-5	8260B	58	RIDEM	19.3	1.0	0.50	0.15
1,3-Dichlorobenzene	541-73-1	8260B	8.7	RIDEM	2.9	1.0	0.50	0.13
1,4-Dichlorobenzene	106-46-7	8260B	1.2	RIDEM	0.4	1.0	0.50	0.12
2-Butanone	78-93-3	8260B	14000	SCV	4667	5.0	1.0	0.55
2-Hexanone	591-78-6	8260B	99	SCV	33.0	5.0	1.0	0.29
4-Methyl-2-pentanone	108-10-1	8260B	170	SCV	56.7	5.0	0.50	0.28
Acetone	67-64-1	8260B	1500	SCV	500.0	10.0	5.0	2.1
Benzene	71-43-2	8260B	5.9	RIDEM	2.0	1.0	0.50	0.18
Bromodichloromethane	75-27-4	8260B	11000	NOAA	3667	1.0	0.50	0.13
Bromoform	75-25-2	8260B	33	RIDEM	11.0	1.0	0.50	0.15
Bromomethane	74-83-9	8260B	--	--	--	--[4]	--[4]	--[4]

Surface Water Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Carbon disulfide	**75-15-0**	**8260B**	**0.92**	**SCV**	**0.3**	**5.0**	**0.50**	**0.18**
Carbon tetrachloride	56-23-5	8260B	30	RIDEM	10.0	1.0	0.50	0.13
Chlorobenzene	108-90-7	8260B	18	RIDEM	6.0	1.0	0.50	0.19
Chloroethane	75-00-3	8260B	--	--	--	1.0	0.50	0.15
Chloroform	67-66-3	8260B	32	RIDEM	10.7	1.0	0.50	0.17
Chloromethane	74-87-3	8260B	--	--	--	1.0	0.50	0.16
cis-1,2-Dichloroethene	156-59-2	8260B	590	SCV	196.7	1.0	0.50	0.23
cis-1,3-Dichloropropene	**10061-01-5**	**8260B**	**0.055**	**SCV**	**0.02**	**1.0**	**0.50**	**0.25**
Cyclohexane	110-82-7	8260B	--	--	--	5.0	0.50	0.25
Dibromochloromethane	124-48-1	8260B	11000	NOAA	3667	1.0	0.50	0.14
Dichlorodifluoromethane	75-71-8	8260B	--		--	1.0	0.50	0.21
Ethylbenzene	100-41-4	8260B	36	RIDEM	12.0	1.0	0.50	0.14
Isopropylbenzene	98-82-8	8260B	--	--	--	1.0	0.50	0.15
Methyl acetate	79-20-9	8260B	--	--	--	5.0	0.50	0.30
Methylcyclohexane	108-87-2	8260B	--	--	--	5.0	0.50	0.20
Methylene chloride	75-09-2	8260B	214	RIDEM	71.3	1.0	0.50	0.26
Methyl-tert-butyl ether	1634-04-4	8260B	10000	NOAA	3333	1.0	0.50	0.28
Styrene	100-42-5	8260B	72	NOAA	24.0	1.0	0.50	0.11
Tetrachloroethene	127-18-4	8260B	5.3	RIDEM	1.8	1.0	0.50	0.16
Toluene	108-88-3	8260B	14	RIDEM	4.7	1.0	0.50	0.16
trans-1,2-Dichloroethene	156-60-5	8260B	590	SCV	196.7	1.0	0.50	0.20
trans-1,3-Dichloropropene	10061-02-6	**8260B**	**0.055**	**SCV**	**0.02**	**1.0**	**0.50**	**0.23**
Trichloroethene	79-01-6	8260B	43	RIDEM	14.3	1.0	0.25	0.092
Trichlorofluoromethane	75-69-4	8260B	11000	NOAA	3667	1.0	0.50	0.20
Vinyl chloride	75-01-4	8260B	930	NOAA	310.0	1.0	0.25	0.10
Xylenes (total)	1330-20-7	8260B	3	RIDEM	1.0	3.0	1.0	0.42

Surface Water Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
PAHs (ug/L)								
2-Methylnaphthalene	91-57-6	8270C SIM	30	NOAA	10.0	0.020	0.0040	0.0040
Acenaphthene	83-32-9	8270C SIM	1	RIDEM	0.3	0.020	0.0040	0.0040
Acenaphthylene	208-96-8	8270C SIM	484	NOAA	161.3	0.020	0.0040	0.0040
Anthracene	120-12-7	8270C SIM	0.73	SCV	0.2	0.020	0.0040	0.0040
Benzo(a)anthracene	56-55-3	8270C SIM	0.027	SCV	0.01	0.020	0.0040	0.0040
Benzo(a)pyrene	**50-32-8**	**8270C SIM**	**0.014**	**SCV**	**0.005**	**0.020**	**0.0040**	**0.0040**
Benzo(b)fluoranthene	205-99-2	8270C SIM	0.9	NOAA	0.3	0.020	0.0040	0.0040
Benzo(g,h,i)perylene	191-24-2	8270C SIM	0.76	NOAA	0.3	0.020	0.0040	0.0040
Benzo(k)fluoranthene	207-08-9	8270C SIM	0.6415	FCV	0.2	0.020	0.0040	0.0040
Chrysene	218-01-9	8270C SIM	2.042	FCV	0.7	0.020	0.0040	0.0040
Dibenzo(a,h)anthracene	53-70-3	8270C SIM	0.2825	FCV	0.1	0.020	0.0040	0.0040
Fluoranthene	206-44-0	8270C SIM	4.4	NOAA	1.5	0.020	0.0040	0.0040
Fluorene	86-73-7	8270C SIM	3.9	SCV	1.3	0.020	0.0040	0.0040
Indeno(1,2,3-cd)pyrene	193-39-5	8270C SIM	4.31	NOAA	1.4	--[4]	--[4]	--[4]
Naphthalene	91-20-3	8270C SIM	2.6	RIDEM	0.9	0.020	0.0040	0.0040
Phenanthrene	85-01-8	8270C SIM	6.3	SCV	2.1	0.020	0.0040	0.0040
Pyrene	129-00-0	8270C SIM	0.025	NOAA	0.01	0.020	0.0040	0.0040
Pesticides (ug/L)								
4,4'-DDE	72-55-9	8081A	105	NOAA	35.0	0.010	0.0008	0.00072
4,4'-DDD	72-54-8	8081A	0.011	SCV	0.004	0.010	0.0008	0.00058
4,4'-DDT	**50-29-3**	**8081A**	**0.001**	**RIDEM**	**0.0003**	**0.010**	**0.0008**	**0.00065**
Aldrin	309-00-2	8081A	3	NRWQC	1.0	0.010	0.0008	0.00080
alpha-BHC	319-84-6	8081A	2.2	SCV	0.7	0.010	0.0008	0.00063
alpha-Chlordane	**5103-71-9**	**8081A**	**0.0043**	**NRWQC**	**0.0**	**0.010**	**0.0008**	**0.00052**
beta-BHC	319-85-7	8081A	2.2	SCV	0.7	0.010	0.0008	0.00052
delta-BHC	319-86-8	8081A	2.2	SCV	0.7	0.010	0.0008	0.0011

Surface Water Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Dieldrin	60-57-1	8081A	0.056	NRWQC	0.02	0.010	0.0008	0.00048
Endosulfan I	959-98-8	8081A	0.056	NRWQC	0.02	0.010	0.0008	0.00046
Endosulfan II	33213-65-9	8081A	0.056	NRWQC	0.02	0.010	0.0008	0.00045
Endosulfan sulfate	1031-07-8	8081A	0.051	SCV	0.02	0.010	0.0008	0.00076
Endrin	72-20-8	8081A	0.036	NRWQC	0.01	0.010	0.0008	0.0062
Endrin aldehyde	7421-93-4	8081A	0.15	NOAA	0.1	0.010	0.008	0.0039
Endrin ketone	53494-70-5	8081A	--	--	--	0.010	0.008	0.00093
gamma-BHC (Lindane)	58-89-9	8081A	0.08	NOAA	0.03	0.010	0.0008	0.00064
gamma-Chlordane	**5103-74-2**	**8081A**	**0.0043**	**NRWQC**	**0.001**	**0.010**	**0.0008**	**0.00046**
Heptachlor	**76-44-8**	**8081A**	**0.0038**	**NRWQC**	**0.001**	**0.010**	**0.0008**	**0.00043**
Heptachlor epoxide	**1024-57-3**	**8081A**	**0.0038**	**NRWQC**	**0.001**	**0.010**	**0.0008**	**0.00056**
Methoxychlor	72-43-5	8081A	0.019	SCV	0.006	0.010	0.008	0.0014
Toxaphene	**8001-35-2**	**8081A**	**0.0002**	**NRWQC**	**0.000**	**0.10**	**0.033**	**0.0087**
PCBs (ug/L)								
Aroclor-1016	**12674-11-2**	**8082**	**0.014**	**NRWQC**	**0.005**	**0.10**	**0.09**	**0.025**
Aroclor-1221	**11104-28-2**	**8082**	**0.014**	**NRWQC**	**0.005**	**0.10**	**0.12**	**0.029**
Aroclor-1232	**11141-16-5**	**8082**	**0.014**	**NRWQC**	**0.005**	**0.10**	**0.08**	**0.032**
Aroclor-1242	**53469-21-9**	**8082**	**0.014**	**NRWQC**	**0.005**	**0.10**	**0.10**	**0.040**
Aroclor-1248	**12672-29-6**	**8082**	**0.014**	**NRWQC**	**0.005**	**0.10**	**0.10**	**0.030**
Aroclor-1254	**11097-69-1**	**8082**	**0.014**	**NRWQC**	**0.005**	**0.10**	**0.08**	**0.033**
Aroclor-1260	**11096-82-5**	**8082**	**0.014**	**NRWQC**	**0.005**	**0.10**	**0.09**	**0.026**

Surface Water Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Total Polychlorinated Biphenyls	**1336-36-3**		**0.014**	**NRWQC**	**0.005**	**0.10**	**0.10**	**0.040**
Metals (ug/L)								
Aluminum	7429-90-5	6010C	87	NRWQC	29.0	50	5.0	23
Antimony	7440-36-0	6020A	10	RIDEM	3.3	2.0	1.0	0.36
Arsenic	7440-38-2	6020A	150	NRWQC	50.0	5.0	1.0	0.38
Barium	7440-39-3	6020A	4	SCV	1.3	2.0	1.0	0.59
Beryllium	**7440-41-7**	**6020A**	**0.17**	**RIDEM**	**0.06**	**1.0**	**1.0**	**0.30**
Cadmium	7440-43-9	6020A	0.25	NRWQC	0.08	0.20	0.20	0.055
Calcium	7440-70-2	6010C	--	--	--	500	100	67
Chromium	7440-47-3	6020A	11	NRWQC	3.7	1.0	1.0	0.46
Cobalt	7440-48-4	6020A	23	SCV	7.7	1.0	0.10	0.042
Copper	7440-50-8	6020A	9	NRWQC	3.0	1.0	1.0	0.71
Iron	7439-89-6	6010C	1000	NRWQC	333.3	20	10	4.2
Lead	7439-92-1	6020A	2.5	NRWQC	0.8	1.0	0.50	0.22
Magnesium	7439-95-4	6010C	--	--	--	500	100	48
Manganese	7439-96-5	6020A	120	SCV	40.0	2.0	1.0	0.42
Mercury	7439-97-6	7470A	0.77	NRWQC	0.3	0.20	0.10	0.061
Nickel	7440-02-0	6020A	52	NRWQC	17.3	1.0	1.0	0.37
Potassium	7440-09-7	6010C	--	--	--	500	100	82
Selenium	7782-49-2	6020A	5	NRWQC	1.7	5.0	1.0	0.82
Silver	**7440-22-4**	**6020A**	**0.36**	**SCV**	**0.1**	**0.50**	**0.20**	**0.061**
Sodium	7440-23-5	6010C	--	--	--	500	250	140
Thallium	7440-28-0	6020A	1	RIDEM	0.3	0.20	0.20	0.056
Vanadium	7440-62-2	6020A	20	SCV	6.7	5.0	1.0	0.28
Zinc	7440-66-6	6020A	120	NRWQC	40.0	10	4.0	2.9

-- = Not available or not applicable.
1. All methods follow SW-846.
2. NRWQC = National Recommended Water Quality Criteria (USEPA, 2009)

RIDEM = Rhode Island Department of Environmental Management (RIDEM, 2009)
SCV = Secondary Chronic Values (Suter and Tsao, 1996)
BTAG = EPA Region III BTAG, Freshwater Screening Benchmarks (USEPA, 2006)
NOAA = NOAA Screening Quick Reference Tables (Buchman, 2008)
FCV=Final Chronic Values (USEPA, 2003c)

3. Laboratory-specific LOQ, LODs, and MDLs are limits that Trimatrix Laboratory achieves when performing a specific analytical method.
4. Laboratory-specific LOQ, LODs, and MDLs limits for bromomethane and indeno(1,2,3-cd)pyrene have not been provided by Trimatrix Laboratory but will be added when available.

Bold rows indicate that the PAL is less than the corresponding QL but greater than the corresponding MDL.
Bold and shaded rows indicate that the PAL is less than the corresponding QL and the MDL.

SAP Worksheet #15c – Soil Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil Samples

Soil Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
VOCs (mg/kg)								
1,1,1-Trichloroethane	71-55-6	8260B	0.07	TV	0.02	0.002	0.001	0.00035
1,1,2,2-Tetrachloroethane	79-34-5	8260B	0.56	RSL	0.2	0.002	0.00025	0.00014
1,1,2-Trichloroethane	79-00-5	8260B	0.4	TV	0.1	0.002	0.001	0.00027
1,1-Dichloroethane	75-34-3	8260B	0.02	TV	0.01	0.002	0.001	0.00025
1,1-Dichloroethene	75-35-4	8260B	0.1	TV	0.03	0.002	0.001	0.00035
1,2,4-Trichlorobenzene	120-82-1	8260B	4.9	RSL	6.7	0.002	0.001	0.00023
1,2-Dibromoethane (EDB)	106-93-4	8260B	0.034	RSL	0.01	0.003	0.0005	0.00015
1,2-Dibromo-3-chloropropane (DBCP)	**96-12-8**	**8260B**	**0.0054**	**RSL**	**0.002**	**0.010**	**0.005**	**0.005**
1,2-Dichlorobenzene	95-50-1	8260B	0.03	TV	0.01	0.001	0.001	0.00015
1,2-Dichloroethane	107-06-2	8260B	0.02	TV	0.01	0.001	0.00025	0.00022
1,2-Dichloropropane	78-87-5	8260B	0.89	RSL	0.3	0.003	0.0005	0.00021
1,3-Dichlorobenzene	541-73-1	8260B	0.03	TV	0.01	0.001	0.0005	0.00015
1,4-Dichlorobenzene	106-46-7	8260B	2.4	RSL	0.8	0.001	0.0005	0.00013
2-Butanone	78-93-3	8260B	2800	RSL	933.3	0.010	0.0005	0.00039
2-Hexanone	591-78-6	8260B	21	RSL	7.0	0.010	0.0005	0.00022
4-Methyl-2-pentanone	108-10-1	8260B	530	RSL	177	0.010	0.001	0.00030
Acetone	67-64-1	8260B	6,100	RSL	2033	0.015	0.005	0.0021
Benzene	71-43-2	8260B	1.1	RSL	0.4	0.001	0.0005	0.00017
Bromodichloromethane	75-27-4	8260B	0.27	RSL	0.1	0.001	0.0005	0.00021
Bromoform	75-25-2	8260B	61	RSL	20.3	0.002	0.0005	0.00017
Bromomethane	74-83-9	8260B	0.73	RSL	0.2	--[4]	--[4]	--[4]

Soil Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Carbon disulfide	75-15-0	8260B	82	RSL	27.3	0.001	0.001	0.00050
Carbon tetrachloride	56-23-5	8260B	0.61	RSL	0.2	0.002	0.001	0.00033
Chlorobenzene	108-90-7	8260B	29	RSL	9.7	0.001	0.0005	0.00017
Chloroethane	75-00-3	8260B	1500	RSL	500.0	0.002	0.001	0.00034
Chloroform	67-66-3	8260B	0.02	TV	0.01	0.001	0.001	0.00051
Chloromethane	74-87-3	8260B	12	RSL	4.0	0.001	0.001	0.00027
cis-1,2-Dichloroethene	156-59-2	8260B	0.2	TV	0.1	0.001	0.001	0.00030
cis-1,3-Dichloropropene	10061-01-5	8260B	1.7	RSL	0.6	0.001	0.0005	0.00020
Cyclohexane	110-82-7	8260B	700	RSL	233.3	0.005	0.001	0.00031
Dibromochloromethane	124-48-1	8260B	0.68	RSL	0.2	0.001	0.0005	0.00012
Dichlorodifluoromethane	75-71-8	8260B	18	RSL	6.0	0.002	0.001	0.00042
Ethylbenzene	100-41-4	8260B	1.2	CCME	0.4	0.001	0.0005	0.00022
Isopropylbenzene	98-82-8	8260B	210	RSL	70	0.001	0.001	0.00029
Methyl acetate	79-20-9	8260B	7800	RSL	2600	0.005	0.001	0.00042
Methylcyclohexane	108-87-2	8260B	--	--	--	0.005	0.001	0.00042
Methylene chloride	75-09-2	8260B	0.4	TV	0.1	0.015	0.001	0.00070
Methyl-tert-butyl ether	1634-04-4	8260B	43	RSL	14.3	0.001	0.001	0.00030
Styrene	100-42-5	8260B	300	ORNL	100.0	0.001	0.0005	0.00022
Tetrachloroethene	127-18-4	8260B	0.55	RSL	0.2	0.002	0.001	0.00028
Toluene	108-88-3	8260B	200	ORNL	66.7	0.002	0.001	0.00027
trans-1,2-Dichloroethene	156-60-5	8260B	15	RSL	5.0	0.001	0.001	0.00033
trans-1,3-Dichloropropene	10061-02-6	8260B	1.7	RSL	0.6	0.003	0.001	0.00025
Trichloroethene	79-01-6	8260B	2.8	RSL	0.9	0.002	0.001	0.00025
Trichlorofluoromethane	75-69-4	8260B	79	RSL	26.3	0.002	0.001	0.00028
Vinyl chloride	75-01-4	8260B	0.01	TV	0.003	0.002	0.001	0.00027
Xylenes (total)	1330-20-7	8260B	1	CCME	0.3	0.003	0.0015	0.00065

Soil Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
PAHs (mg/kg)								
2-Methylnaphthalene	91-57-6	8270C SIM	29	Eco SSL	10.3	0.0020	0.00013	0.00013
Acenaphthene	83-32-9	8270C SIM	29	Eco SSL	9.7	0.0020	0.00013	0.00013
Acenaphthylene	208-96-8	8270C SIM	293	Eco SSL	113	0.0020	0.00013	0.00013
Anthracene	120-12-7	8270C SIM	29	Eco SSL	9.7	0.0020	0.00013	0.00013
Benzo(a)anthracene	56-55-3	8270C SIM	0.15	RSL	0.1	0.0020	0.00013	0.00013
Benzo(a)pyrene	50-32-8	8270C SIM	0.015	RSL	0.01	0.0020	0.00013	0.00013
Benzo(b)fluoranthene	205-99-2	8270C SIM	0.15	RSL	0.1	0.0020	0.00013	0.00013
Benzo(g,h,i)perylene	191-24-2	8270C SIM	1.1	Eco SSL	0.4	0.0020	0.00013	0.00013
Benzo(k)fluoranthene	207-08-9	8270C SIM	1.1	Eco SSL	0.4	0.0020	0.00013	0.00013
Chrysene	218-01-9	8270C SIM	1.1	Eco SSL	0.4	0.0020	0.00013	0.00013
Dibenzo(a,h)anthracene	53-70-3	8270C SIM	0.015	RSL	0.01	0.0020	0.00013	0.00013
Fluoranthene	206-44-0	8270C SIM	29	Eco SSL	9.7	0.0020	0.00013	0.00013
Fluorene	86-73-7	8270C SIM	29	Eco SSL	9.7	0.0020	0.00013	0.00013
Indeno(1,2,3-cd)pyrene	193-39-5	8270C SIM	0.15	RSL	0.1	--[4]	--[4]	--[4]
Naphthalene	91-20-3	8270C SIM	3.6	RSL	1.2	0.0020	0.00013	0.00013
Phenanthrene	85-01-8	8270C SIM	29	Eco SSL	9.7	0.0020	0.00013	0.00013
Pyrene	129-00-0	8270C SIM	1.1	Eco SSL	0.4	0.0020	0.00013	0.00013
Pesticides (mg/kg)								
4,4'-DDE	72-55-9	8081A	1.4	RSL	0.5	0.017	0.00013	0.000028
4,4'-DDD	72-54-8	8081A	2	RSL	0.7	0.017	0.0013	0.00030
4,4'-DDT	50-29-3	8081A	0.021	Eco SSL	0.01	0.017	0.0013	0.00029
Aldrin	**309-00-2**	**8081A**	**0.00006**	**TV**	**0.00002**	**0.017**	**0.0013**	**0.0013**
alpha-BHC	**319-84-6**	**8081A**	**0.003**	**TV**	**0.001**	**0.017**	**0.00013**	**0.000054**
alpha-Chlordane	**5103-71-9**	**8081A**	**0.00003**	**TV**	**0.00001**	**0.017**	**0.0013**	**0.00040**
beta-BHC	**319-85-7**	**8081A**	**0.009**	**TV**	**0.003**	**0.017**	**0.0013**	**0.00029**

Soil Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
delta-BHC	319-86-8	**8081A**	**0.01**	**TV**	**0.003**	**0.017**	**0.0013**	**0.00027**
Dieldrin	**60-57-1**	**8081A**	**0.0049**	**Eco SSL**	**0.002**	**0.017**	**0.00013**	**0.000035**
Endosulfan I	959-98-8	**8081A**	**0.00001**	**TV**	**0.000003**	**0.017**	**0.00013**	**0.000015**
Endosulfan II	33213-65-9	**8081A**	**0.00001**	**TV**	**0.000003**	**0.017**	**0.0013**	**0.00027**
Endosulfan sulfate	**1031-07-8**	**8081A**	**0.00001**	**TV**	**0.000003**	**0.017**	**0.0013**	**0.00033**
Endrin	**72-20-8**	**8081A**	**0.00004**	**TV**	**0.00001**	**0.017**	**0.0013**	**0.00028**
Endrin aldehyde	**7421-93-4**	**8081A**	**0.00004**	**TV**	**0.00001**	**0.017**	**0.0013**	**0.00091**
Endrin ketone	**53494-70-5**	**8081A**	**0.00004**	**TV**	**0.00001**	**0.017**	**0.0013**	**0.00036**
gamma-BHC (Lindane)	**58-89-9**	**8081A**	**0.00005**	**TV**	**0.00002**	**0.017**	**0.00013**	**0.000040**
gamma-Chlordane	**5103-74-2**	**8081A**	**0.00003**	**TV**	**0.00001**	**0.017**	**0.0013**	**0.00028**
Heptachlor	**76-44-8**	**8081A**	**0.0007**	**TV**	**0.0002**	**0.017**	**0.0013**	**0.00043**
Heptachlor epoxide	**1024-57-3**	**8081A**	**0.0000002**	**TV**	**0.0000001**	**0.017**	**0.00013**	**0.000035**
Methoxychlor	72-43-5	8081A	31	RSL	10.3	0.017	0.0013	0.00040
Toxaphene	8001-35-2	8081A	0.44	RSL	0.1	0.17	0.0033	0.0029
PCBs (mg/kg)								
Aroclor-1016	12674-11-2	8082	0.39	RSL	0.13	0.033	0.010	0.0042
Aroclor-1221	11104-28-2	8082	0.14	RSL	0.05	0.033	0.020	0.0078
Aroclor-1232	11141-16-5	8082	0.14	RSL	0.05	0.033	0.010	0.0045
Aroclor-1242	53469-21-9	8082	0.22	RSL	0.1	0.033	0.010	0.0046
Aroclor-1248	12672-29-6	8082	0.22	RSL	0.1	0.033	0.017	0.0065
Aroclor-1254	11097-69-1	8082	0.22	RSL	0.1	0.033	0.013	0.0059

Soil Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Aroclor-1260	11096-82-5	8082	0.22	RSL	0.1	0.033	0.010	0.0050
Total Polychlorinated Biphenyls	1336-36-3		--	--	0.13	0.033	0.010	0.0078
GRO and DRO (mg/kg)								
GRO (C6-C10)	NA	8015B	500	RIDEM TPH	166.7	5.0	4.0	1.2
DRO (C8-C40)	NA	8015B	500	RIDEM TPH	166.7	10	3.3	1.3
Metals (mg/kg)								
Aluminum	7429-90-5	6010C	7700	RSL	2567	10	5.0	2.7
Antimony	7440-36-0	6020A	0.27	Eco SSL	0.1	0.20	0.10	0.045
Arsenic	**7440-38-2**	**6020A**	**0.39**	**RSL**	**0.1**	**0.50**	**0.10**	**0.039**
Barium	7440-39-3	6020A	330	Eco SSL	110.0	0.10	0.05	0.021
Beryllium	7440-41-7	6020A	16	RSL	5.3	0.10	0.10	0.031
Cadmium	7440-43-9	6020A	0.36	Eco SSL	0.1	0.050	0.02	0.0076
Calcium	7440-70-2	6010C	--	--	--	50	9.0	6.8
Chromium	7440-47-3	6020A	0.29	RSL	0.1	0.20	0.10	0.057
Cobalt	7440-48-4	6020A	2.3	RSL	0.8	0.10	0.01	0.0080
Copper	7440-50-8	6020A	28	Eco SSL	9.3	0.20	0.10	0.078
Iron	7439-89-6	6010C	5500	RSL	1833	5.0	1.0	0.47
Lead	7439-92-1	6020A	11	Eco SSL	3.7	0.10	0.10	0.054
Magnesium	7439-95-4	6010C	--	--	--	50	10	4.9
Manganese	7439-96-5	6020A	180	RSL	60.0	0.20	0.10	0.079
Mercury	**7439-97-6**	**7471A**	**0.00051**	**ORNL**	**0.00**	**0.050**	**0.017**	**0.016**
Nickel	7440-02-0	6020A	38	Eco SSL	12.7	0.20	0.10	0.030
Potassium	7440-09-7	6010C	--	--	--	50	10	7.2
Selenium	7782-49-2	6020A	0.52	Eco SSL	0.2	0.50	0.10	0.088
Silver	7440-22-4	6020A	4.2	Eco SSL	1.4	0.10	0.02	0.011

Soil Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Sodium	7440-23-5	6010C	--	--	--	50	20	5.2
Thallium	7440-28-0	6020A	1	ORNL	0.3	0.10	0.02	0.016
Vanadium	7440-62-2	6020A	0.55	RSL	0.2	0.10	0.10	0.040
Zinc	7440-66-6	6020A	46	Eco SSL	15.3	2.0	0.50	0.46

-- = Not available or not applicable.

1. All methods follow SW-846.
2.
   RIDEM TPH Res = Rhode Island Department of Environmental Management Residential TPH Direct Exposure Criteria (RIDEM, 2004)
   CCME = Canadian Council of Ministers of the Environment (CCME, 1997)
   Eco SSL = Soil Screening Level (USEPA, 2003, 2005, 2006, 2007, 2008)
   RSL = EPA RSLs for Chemical Contaminants at Superfund Sites, Residential Sol Values (EPA 2009). One-tenth RSLs are displayed for noncancer RSLs to represent a target hazard quotient of 0.1.
   ORNL=Oak Ridge National Laboratory (Efroymson, et al, 1997a, 1997b)
3. Laboratory-specific LOQ, LODs, and MDLs are limits that Trimatrix Laboratory can achieve when performing a specific analytical method.
4. Laboratory-specific LOQ, LODs, and MDLs limits for bromomethane and indeno(1,2,3-cd)pyrene have not been provided by Trimatrix Laboratory but will be added when available.

Bold rows indicate that the PAL is less than the corresponding QL but greater than the corresponding MDL.
Bold and shaded rows indicate that the PAL is less than the corresponding QL and the MDL.

SAP Worksheet #15d – Sediment Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Sediment Samples

Sediment Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
VOCs (ug/kg)								
1,1,1-Trichloroethane	71-55-6	8260B	170	EPA SQB	56.7	2.0	1.0	0.35
1,1,2,2-Tetrachloroethane	79-34-5	8260B	940	EPA SQB	313.3	2.0	0.25	0.14
1,1,2-Trichloroethane	79-00-5	8260B	1200	SCV	400.0	2.0	1.0	0.27
1,1-Dichloroethane	75-34-3	8260B	27	SCV	9.0	2.0	1.0	0.25
1,1-Dichloroethene	75-35-4	8260B	31	SCV	10.3	2.0	1.0	0.35
1,2,4-Trichlorobenzene	120-82-1	8260B	9200	EPA SQB	3067	2.0	1.0	0.23
1,2-Dibromoethane (EDB)	106-93-4	8260B	--	--	--	3.0	0.50	0.15
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	8260B	--	--	--	10.0	5.0	5
1,2-Dichlorobenzene	95-50-1	8260B	340	EPA SQB	113.3	1.0	1.0	0.15
1,2-Dichloroethane	107-06-2	8260B	250	SCV	83.3	1.0	0.25	0.22
1,2-Dichloropropane	78-87-5	8260B	--	--	--	3.0	0.50	0.21
1,3-Dichlorobenzene	541-73-1	8260B	1700	EPA SQB	566.7	1.0	0.50	0.15
1,4-Dichlorobenzene	106-46-7	8260B	350	EPA SQB	116.7	1.0	0.50	0.13
2-Butanone	78-93-3	8260B	270	SCV	90.0	10.0	0.50	0.39
2-Hexanone	591-78-6	8260B	22	SCV	7.3	10.0	0.50	0.22
4-Methyl-2-pentanone	108-10-1	8260B	33	SCV	11.0	10.0	1.0	0.30
Acetone	**67-64-1**	**8260B**	**9**	**SCV**	**3.0**	**15.0**	**5.0**	**2.1**
Benzene	71-43-2	8260B	57	EPA SQB	19.0	1.0	0.50	0.17
Bromodichloromethane	75-27-4	8260B	--	--	--	1.0	0.50	0.21
Bromoform	75-25-2	8260B	650	EPA SQB	216.7	2.0	0.50	0.17
Bromomethane	74-83-9	8260B	1.37	ESV	0.47	--[4]	--[4]	--[4]

Sediment Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Carbon disulfide	**75-15-0**	**8260B**	**0.85**	**SCV**	**0.3**	**1.0**	**1.0**	**0.50**
Carbon tetrachloride	56-23-5	8260B	1200	EPA SQB	400.0	2.0	1.0	0.33
Chlorobenzene	108-90-7	8260B	820	EPA SQB	273.3	1.0	0.50	0.17
Chloroethane	75-00-3	8260B	--	--	--	2.0	1.0	0.34
Chloroform	67-66-3	8260B	22	SCV	7.3	1.0	1.0	0.51
Chloromethane	74-87-3	8260B	--	--	--	1.0	1.0	0.27
cis-1,2-Dichloroethene	156-59-2	8260B	400	SCV	133.3	1.0	1.0	0.30
cis-1,3-Dichloropropene	10061-01-5	**8260B**	**0.051**	**SCV**	**0.02**	**1.0**	**0.50**	**0.20**
Cyclohexane	110-82-7	8260B	--	--	--	5.0	1.0	0.31
Dibromochloromethane	124-48-1	8260B	--	--	--	1.0	0.50	0.12
Dichlorodifluoromethane	75-71-8	8260B	--	--	--	2.0	1.0	0.42
Ethylbenzene	100-41-4	8260B	3600	EPA SQB	1200	1.0	0.50	0.22
Isopropylbenzene	98-82-8	8260B	--	--	--	1.0	1.0	0.29
Methyl acetate	79-20-9	8260B	--	--	--	5.0	1.0	0.42
Methylcyclohexane	108-87-2	8260B	--	--	--	5.0	1.0	0.42
Methylene chloride	75-09-2	8260B	370	SCV	123.3	15.0	1.0	0.70
Methyl-tert-butyl ether	1634-04-4	8260B	--	--	--	1.0	1.0	0.30
Styrene	100-42-5	8260B	--	--	--	1.0	0.50	0.22
Tetrachloroethene	127-18-4	8260B	530	EPA SQB	176.7	2.0	1.0	0.28
Toluene	108-88-3	8260B	670	EPA SQB	223.3	2.0	1.0	0.27
trans-1,2-Dichloroethene	156-60-5	8260B	400	SCV	133.3	1.0	1.0	0.33
trans-1,3-Dichloropropene	10061-02-6	**8260B**	**0.051**	**SCV**	**0.02**	**3.0**	**1.0**	**0.25**
Trichloroethene	79-01-6	8260B	1600	EPA SQB	533.3	2.0	1.0	0.25
Trichlorofluoromethane	75-69-4	8260B	--	--	--	2.0	1.0	0.28
Vinyl chloride	75-01-4	8260B	--	--	--	2.0	1.0	0.27
Xylenes (total)	1330-20-7	8260B	130	SCV	43.3	3.0	1.5	0.65

Sediment Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
PAHs (ug/kg)								
2-Methylnaphthalene	91-57-6	8270C SIM	20.2	ESV	6.7	2.0	0.13	0.13
Acenaphthene	83-32-9	8270C SIM	290	NOAA	96.7	2.0	0.13	0.13
Acenaphthylene	208-96-8	8270C SIM	160	NOAA	53.3	2.0	0.13	0.13
Anthracene	120-12-7	8270C SIM	57.2	TEC	19.1	2.0	0.13	0.13
Benzo(a)anthracene	56-55-3	8270C SIM	108	TEC	36.0	2.0	0.13	0.13
Benzo(a)pyrene	50-32-8	8270C SIM	150	TEC	50.0	2.0	0.13	0.13
Benzo(b)fluoranthene	205-99-2	8270C SIM	1800	NOAA	600.0	2.0	0.13	0.13
Benzo(g,h,i)perylene	191-24-2	8270C SIM	170	LEL	56.7	2.0	0.13	0.13
Benzo(k)fluoranthene	207-08-9	8270C SIM	240	LEL	80.0	2.0	0.13	0.13
Chrysene	218-01-9	8270C SIM	166	TEC	55.3	2.0	0.13	0.13
Dibenzo(a,h)anthracene	53-70-3	8270C SIM	33	TEC	11.0	2.0	0.13	0.13
Fluoranthene	206-44-0	8270C SIM	423	TEC	141.0	2.0	0.13	0.13
Fluorene	86-73-7	8270C SIM	77.4	TEC	25.8	2.0	0.13	0.13
Indeno(1,2,3-cd)pyrene	193-39-5	8270C SIM	200	LEL	66.67	--[4]	--[4]	--[4]
Naphthalene	91-20-3	8270C SIM	176	TEC	58.7	2.0	0.13	0.13
Phenanthrene	85-01-8	8270C SIM	204	TEC	68.0	2.0	0.13	0.13
Pyrene	129-00-0	8270C SIM	195	TEC	65.0	2.0	0.13	0.13
Pesticides (ug/kg)								
4,4'-DDE	**72-55-9**	**8081A**	**3.16**	**TEC**	**1.1**	**17**	**0.13**	**0.028**
4,4'-DDD	**72-54-8**	**8081A**	**4.88**	**TEC**	**1.6**	**17**	**1.3**	**0.30**
4,4'-DDT	**50-29-3**	**8081A**	**4.16**	**TEC**	**1.4**	**17**	**1.3**	**0.29**
Aldrin	**309-00-2**	**8081A**	**2**	**LEL**	**0.7**	**17**	**1.3**	**1.3**
alpha-BHC	**319-84-6**	**8081A**	**6**	**LEL**	**2.0**	**17**	**0.13**	**0.054**
alpha-Chlordane	**5103-71-9**	**8081A**	**3.24**	**TEC**	**1.1**	**17**	**1.3**	**0.40**
beta-BHC	**319-85-7**	**8081A**	**5**	**LEL**	**1.7**	**17**	**1.3**	**0.29**

Sediment Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
delta-BHC	319-86-8	8081A	120	SCV	40.0	17	1.3	0.27
Dieldrin	**60-57-1**	**8081A**	**2**	**LEL**	**0.7**	**17**	**0.13**	**0.035**
Endosulfan I	959-98-8	**8081A**	**2.9**	**EPA SQB**	**1.0**	**17**	**0.13**	**0.015**
Endosulfan II	33213-65-9	**8081A**	**14**	**SQB**	**4.7**	**17**	**1.3**	**0.27**
Endosulfan sulfate	**1031-07-8**	**8081A**	**5.4**	**EPA SQB**	**1.8**	**17**	**1.3**	**0.33**
Endrin	**72-20-8**	**8081A**	**2**	**TEC**	**0.7**	**17**	**1.3**	**0.28**
Endrin aldehyde	7421-93-4	8081A	--	--	--	17	1.3	0.91
Endrin ketone	53494-70-5	8081A	--	--	--	17	1.3	0.36
gamma-BHC (Lindane)	**58-89-9**	**8081A**	**2.37**	**TEC**	**0.8**	**17**	**0.13**	**0.040**
gamma-Chlordane	**5103-74-2**	**8081A**	**3.24**	**TEC**	**1.1**	**17**	**1.3**	**0.28**
Heptachlor	**76-44-8**	**8081A**	**10**	**NOAA**	**3.3**	**17**	**1.3**	**0.43**
Heptachlor epoxide	**1024-57-3**	**8081A**	**2.47**	**TEC**	**0.8**	**17**	**0.13**	**0.035**
Methoxychlor	72-43-5	8081A	19	EPA SQB	6.3	17	1.3	0.40
Toxaphene	**8001-35-2**	**8081A**	**28**	**EPA SQB**	**9.3**	**170**	**3.3**	**2.9**
PCBs (ug/kg)								
Aroclor-1016	12674-11-2	8082	59.8	TEC	19.9	33	10	4.2
Aroclor-1221	11104-28-2	8082	59.8	TEC	19.9	33	20	7.8
Aroclor-1232	11141-16-5	8082	59.8	TEC	19.9	33	10	4.5
Aroclor-1242	53469-21-9	8082	59.8	TEC	19.9	33	10	4.6
Aroclor-1248	12672-29-6	8082	59.8	TEC	19.9	33	17	6.5
Aroclor-1254	11097-69-1	8082	59.8	TEC	19.9	33	13	5.9

Sediment Samples - Analyte	CAS Number	Analytical Method[1]	Project Action Limit	Project Action Limit Reference[2]	Project Quantitation Limit Goal	TriMatrix Limits[3]		
						LOQ	LOD	MDL
Aroclor-1260	11096-82-5	8082	59.8	TEC	19.9	33	10	5.0
Total Polychlorinated Biphenyls	1336-36-3		59.8	TEC	19.9	33	10	7.8
Metals (mg/kg)								
Aluminum	7429-90-5	6010C	--	--	--	10	5.0	2.7
Antimony	7440-36-0	6020A	3	NOAA	1.0	0.20	0.10	0.045
Arsenic	7440-38-2	6020A	9.79	TEC	3.3	0.50	0.10	0.039
Barium	7440-39-3	6020A	48	NOAA	16.0	0.10	0.05	0.021
Beryllium	7440-41-7	6020A	--	--	--	0.10	0.10	0.031
Cadmium	7440-43-9	6020A	0.99	TEC	0.3	0.050	0.02	0.0076
Calcium	7440-70-2	6010C	--	--	--	50	9.0	6.8
Chromium	7440-47-3	6020A	43.4	TEC	14.5	0.20	0.10	0.057
Cobalt	7440-48-4	6020A	50	NOAA	16.7	0.10	0.01	0.0080
Copper	7440-50-8	6020A	31.6	TEC	10.5	0.20	0.10	0.078
Iron	7439-89-6	6010C	20000	LEL	6667	5.0	1.0	0.47
Lead	7439-92-1	6020A	35.8	TEC	11.9	0.10	0.10	0.054
Magnesium	7439-95-4	6010C	--	--	--	50	10	4.9
Manganese	7439-96-5	6020A	460	LEL	153.3	0.20	0.10	0.079
Mercury	7439-97-6	7471A	0.18	TEC	0.1	0.050	0.017	0.016
Nickel	7440-02-0	6020A	22.7	TEC	7.6	0.20	0.10	0.030
Potassium	7440-09-7	6010C	--	--	--	50	10	7.2
Selenium	7782-49-2	6020A	1	NOAA	0.3	0.50	0.10	0.088
Silver	7440-22-4	6020A	0.5	LEL	0.2	0.10	0.02	0.011
Sodium	7440-23-5	6010C	--	--	--	50	20	5.2
Thallium	7440-28-0	6020A	--	--	--	0.10	0.02	0.016
Vanadium	7440-62-2	6020A	57	NOAA	19.0	0.10	0.10	0.040
Zinc	7440-66-6	6020A	121	TEC	40.3	2.0	0.50	0.46

-- = Not available or not applicable.

1. All methods follow SW-846.
2. RIDEM TPH Res = Rhode Island Department of Environmental Management Residential TPH Direct Exposure Criteria (RIDEM, 2004)
   TEC = Threshold Effects Concentration (MacDonald, et al., 2000)
   LEL = Lowest Effects Level (Persaud, et al., 1993)
   EPA SQB = U.S. Environmental Protection Agency Sediment Quality Benchmark (USEPA, 1996)
   NOAA = National Oceanic and Atmospheric Administration (Buchman, 1999)
   SCV = Secondary Chronic Value (Jones, et al., 1997)

   ESV-Ecological Screening Values (USEPA, 2003)
3. Laboratory-specific LOQ, LODs, and MDLs are limits that Trimatrix Laboratory can achieve when performing a specific analytical method.
4. Laboratory-specific LOQ, LODs, and MDLs limits for bromomethane and indeno(1,2,3-cd)pyrene have not been provided by Trimatrix Laboratory but will be added when available.

Bold rows indicate that the PAL is less than the corresponding QL but greater than the corresponding MDL.
Bold and shaded rows indicate that the PAL is less than the corresponding QL and the MDL.

SAP Worksheet #16 -- Project Schedule / Timeline Table (optional format)
(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
SAP review and finalization	Tetra Tech, Navy, Project Team	August 2010	September 2010	Final (Signed) SAP	Prior to commencing field work
Field activities: mobilization; test pit excavation; soil sampling; well installation; groundwater sampling; sediment/surface water sampling; surveying; demobilization; and IDW management.	Tetra Tech, subcontractors	September 2010	October 2010	NA	NA
Data analysis and reporting: laboratory analysis; data validation; data reduction; and data reporting.	Tetra Tech, subcontractors	October 2010	October 2011	Final SASE Report	October 22, 2011

SAP Worksheet #17 -- Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)

Samples will be collected at the CCRF Area to generate data to support the SASE Investigation. The sampling design and rationale are developed in Worksheet #11 and are summarized below.

17.1 SAMPLING DESIGN AND RATIONALE

The sampling design of this project is based on a need to better define what contaminants are present in the site media and provide data to conduct screening risk assessments.

A judgmental sampling design was chosen for this project. The focus is on the most likely areas to have received chemical releases to the environment, if any, as well as estimated upgradient areas, to evaluate if upgradient conditions may have impacts on the site. The targeted chemical evaluations generally focus on those chemicals that have a reasonable potential to have been released, however, groundwater target analytes include a larger array of chemicals because this medium has not been sampled previously. The design requires advancing soil borings, installing monitoring wells, and completing test pits in the former disposal area of the CCRF Area, with a focus on areas previously identified as having contaminants above state criteria and a focus on areas not previously investigated. In addition, the sample design requires the collection of sediment samples in areas not previously investigated. Surface water samples, which have not been previously collected, will be co-located with each sediment sample location.

17.2 SAMPLING AND ANALYSIS PLAN

The following summarizes the details of the SAP.

Matrices to be Sampled – Overburden groundwater, surficial and subsurface soil samples, sediment, and surface water samples will be collected.

Sampling Frequency/Timing – A single sampling event is anticipated to be performed beginning in September/October 2010.

Sampling Locations/Number of Samples – The sampling locations are presented by media in Tables 17-1, 17-2, and 17-3, and are depicted in Figure 17-1.

Soil Boring and Groundwater Locations – Soil boring and groundwater samples will be collected from eight locations. Borings will be advanced to refusal (depth of borings estimated to be approximately 30 to 40 feet) using drive and wash drilling. The bedrock depth is believed to range from 30 to 40 feet at the

site. Soil will be collected from four intervals at each location, one surface interval, one interval above bedrock, and two intermediate intervals based on field observations. Seven of the monitoring wells will be screened across the water table, which is estimated to be at 8 to 10 feet bgs. One monitoring well (proposed MW05) will be located at former test pit location TP-2 which was identified in the PA investigation as having VOC-contaminated soils; this well will be screened immediately above bedrock. The remaining seven boring/well locations were chosen based on lateral coverage of the fill area.

Field Measurements include the following:

- PID jar-headspace readings will be recorded for each soil boring interval. This information will be used to estimate the potential presence or absence and possible order-of-magnitude of contamination in soil samples (based on elevated PID readings), and will be used by the field geologist to assist in selection of intermediate sample intervals in the soil borings. Elevated PID readings on 10 ppm above background levels will be used as a threshold level to select intervals for sampling.

Test Pit Locations – Advance four test pits, as indicated in Table 17-2 and on Figure 17-1. Test pit locations were selected to further characterize the fill material in areas not previously investigated during the PA.

Sediment and Surface Water Locations – Seven sediment and surface water samples will be collected from the onsite wetland area and stream, as indicated in Table 17-3 and on Figure 17-1. Sample locations were positioned in areas not sampled during the PA investigation. In addition, a suspected fill area in the western portion of the wetland area will be investigated with a sample location.

Number of Samples (excluding field duplicates)

- Groundwater: (one sample per well) x (eight wells) = eight samples
- Soil from soil borings: (four samples per boring location) x (eight borings) = 32 samples
- Soil from test pits: (two samples per test pit) x (four test pits) = eight samples
- Sediment and Surface Water: (one sample per location) x (seven locations) = seven sediment samples and seven surface water samples

Sampling Methods – The sampling methods are listed in Worksheet #18 and discussed in Worksheet #14. Project-specific sampling procedures are presented in Appendix C.

Analytical Groups/Methods -

- Groundwater samples will be analyzed for VOCs, SVOCs, pesticides, PCBs, and TAL metals (total and dissolved).
- Soil, sediment, and surface water samples will be analyzed for VOCs, PAHs, pesticides, PCBs, TAL metals. A subset of soil samples will be analyzed for TPH (GRO/DRO).

The analytical groups and analytical methods are presented in Worksheet #19.

Worksheets #15a through #15d also list analytical methods as well as project action limits and project quantitation limits for analytes. The rationale for the selection of target analytes is discussed in Worksheet #11, Section 11.2.

Table 17-1
Summary of Proposed Borings and Monitoring Wells

Boring/Well ID	Location	Justification
CCRF-SB01/CCRF-MW01	Southwest boundary of site adjacent to residential area	Determine geologic conditions in fill area (confirm the presence of fill, determine depth of fill, depth to bedrock, presence of confining layers, etc.). Determine presence of contaminants in fill. Determine presence of contaminants in groundwater.
CCRF-SB02/CCRF-MW02	Southwest boundary of site adjacent to residential area	Determine geologic conditions in fill area (confirm the presence of fill, determine depth of fill, depth to bedrock, presence of confining layers, etc.). Determine presence of contaminants in fill. Determine presence of contaminants in groundwater and the presence of an upgradient source.
CCRF-SB03/CCRF-MW03	Central southwestern portion of fill area, in between former test pits TP04 and TP05	Determine geologic conditions in fill area (confirm the presence of fill, determine depth of fill, depth to bedrock, presence of confining layers, etc.). Determine presence of contaminants in fill. Determine presence of contaminants in groundwater.
CCRF-SB04/CCRF-MW04	Central portion of fill area	Determine geologic conditions in fill area (confirm the presence of fill, determine depth of fill, depth to bedrock, presence of confining layers, etc.). Determine presence of contaminants in fill. Determine presence of contaminants in groundwater.
CCRF-SB05/CCRF-MW05	Southern boundary of site adjacent to Coddington Cove Highway	Determine geologic conditions in fill area (confirm the presence of fill, determine depth of fill, depth to bedrock, presence of confining layers, etc.). Determine presence of contaminants in fill. Determine presence of contaminants in groundwater and the presence of an upgradient source.
CCRF-SB06/CCRF-MW06	Southern boundary of site adjacent to Coddington Cove Highway	Determine geologic conditions in fill area (confirm the presence of fill, determine depth of fill, depth to bedrock, presence of confining layers, etc.). Determine presence of contaminants in fill. Determine presence of contaminants in groundwater and the presence of an upgradient source.
CCRF-SB07/CCRF-MW07	North central portion of fill area	Determine geologic conditions in fill area (confirm the presence of fill, determine depth of fill, depth to bedrock, presence of confining layers, etc.). Determine presence of contaminants in fill. Determine presence of contaminants in groundwater.
CCRF-SB08/CCRF-MW08	Central portion of fill area	Determine geologic conditions in fill area (confirm the presence of fill, determine depth of fill, depth to bedrock, presence of confining layers, etc.). Determine presence of contaminants in fill. Determine presence of contaminants in groundwater.

Table 17-2
Summary of Proposed Test Pits

Sample ID	Location	Justification
CCRF-TP12	Southwest boundary of site adjacent to residential area	Determine geologic conditions and presence of contaminants in fill area.
CCRF- TP13	Central portion of fill area	Determine geologic conditions and presence of contaminants in fill area.
CCRF- TP14	Central portion of fill area	Determine geologic conditions and presence of contaminants in fill area.
CCRF- TP15	Southern boundary of site adjacent to Coddington Cove Highway	Determine geologic conditions and presence of contaminants in fill area.

Table 17-3
Summary of Proposed Sediment and Surface Water Samples

Sample ID	Location	Justification
CCRF-SD01/SW01	Southwest portion of onsite wetland	Determine presence of contaminants in the onsite wetland area.
CCRF-SD02/SW02	Southwest portion of onsite wetland	Determine presence of contaminants in the onsite wetland area.
CCRF-SD03/SW03	Central portion of onsite wetland	Determine presence of contaminants in the onsite wetland area.
CCRF-SD04/SW04	Northern/downstream portion of stream that borders site to the northeast	Determine presence of contaminants in stream, in downstream area from site.
CCRF-SD05/SW05	North central portion of onsite wetland	Determine presence of contaminants in stream, in central section of stream that borders the site.
CCRF-SD06/SW06	Eastern portion of onsite wetland	Determine presence of contaminants in stream that may be related to upgradient sources (sample near outfall that enters site from the east).
CCRF-SD07/SW07	Eastern portion of onsite wetland	Determine presence of contaminants in eastern portion of onsite wetland area.

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
MW-01 through MW-08	Groundwater	Overburden aquifer	VOCs, SVOCs, pesticides, PCBs, metals.	8 samples, 1 duplicate	Groundwater Sample Acquisition and Onsite Water Quality Testing, SA-1.1 (Revision 7, 4/08)
SB-01 through SB-08[2]	Soil	Four intervals (surface to refusal)	VOCs, PAHs, pesticides, PCBs, metals, GRO/DRO[2]	32 samples, 4[2] duplicates	Soil Sampling, SA-1.3 (Revision 7, 9/03)
TP-12 through TP-15	Soil	Two intervals (surface and subsurface)	VOCs, PAHs, pesticides, PCBs, metals	8 samples, 1 duplicate	Soil Sampling, SA-1.3 (Revision 7, 9/03)
SD-01 through SD-07	Sediment	Surface (0 to 6 inches)	VOCs, PAHs, pesticides, PCBs, metals	7 samples, 1 duplicate	Surface Water and Sediment Sampling, SA-1.2 (Revision 5, 9/03)
SW-01 through SW-07	Surface water	Surface	VOCs, PAHs, pesticides, PCBs, metals	7 samples, 1 duplicate	Surface Water and Sediment Sampling, SA-1.2 (Revision 5, 9/03)

[1]Standard operating procedure (SOP) or worksheet that describes the sample collection procedures.

[2] GRO/DRO analysis will be conducted only at location SB-06

SAP Worksheet #19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)	Sample volume[2] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[3] (preparation / analysis)
Aqueous (Groundwater, Surface Water, and Rinsate Blanks)	VOCs	SW-846 5030/8260B SOP GR-04-104	Three 40-mL VOA vials	40 mL	Hydrochloric acid (HCl) to pH < 2, cool to 4±2 ºC	14 days to analysis
	GRO	SW-846 5030/8015B SOP GR-03-121	Two 40-mL VOA vials	40 mL	HCl to pH < 2, cool to 4±2 ºC	14 days to analysis
	DRO	SW-846 3510C/8015B SOPs: GR-09-124, GR-03-122	Two 1-liter (L) amber glass bottles	1000 mL	Cool to 4±2 ºC	7 days to extraction; 40 days to analysis
	SVOCs or PAHs[4]	SW-846 3510C/8270C or 8270C SIM[5] SOPs: GR-09-101, GR-04-103	Two 1-L amber glass bottles	1000 mL	Cool to 4±2 ºC	7 days to extraction; 40 days to analysis
	Pesticides	SW-846 3510C/8081A SOPs: GR-09-107, GR-03-120	Two 1-L amber glass bottles	1000 mL	Cool to 4±2 ºC	7 days to extraction; 40 days to analysis
	PCBs	SW-846 3510C/8082 SOPs: GR-09-107, GR-03-128	Two 1-L amber glass bottles	1000 mL	Cool to 4±2 ºC	Not applicable
	Total Metals	SW-846 3010A/3020A/6010C/ 6020A/7470A SOPs: GR-01-140, GR-01-147, GR-01-148, GR-01-100, GR-01-123 GR-01-129	500 mL polyethylene	100 mL	Nitric acid (HNO3) to pH < 2, cool to 4±2 ºC	180 days to analysis (ICP metals); 28 days (mercury)
	Dissolved Metals[6]	SW-846 3010A/3020A/6010C/ 6020A/7470A SOPs: GR-01-140, GR-01-147, GR-01-148, GR-01-100, GR-01-123 GR-01-129	500 mL polyethylene	100 mL	HNO3 to pH < 2, cool to 4±2 ºC	180 days to analysis (ICP metals); 28 days (mercury)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)	Sample volume[2] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[3] (preparation / analysis)
Soil and Sediment	VOCs	SW-846 5035A/8260B SOPs: GR-04-105, GR-04-104	Two 5 gram (g) Terracores into pre-preserved tare weighted VOA vial	5 g	1 g $NaHSO_4$ in 5 mL reagent water; cool to 4±2 ºC	14 days to analysis
			One 2 ounce jar (unpreserved) for percent moisture analysis	5 g	Cool to 4±2 ºC	14 days to analysis
			One 5 gram (g) Terracores into pre-preserved tare weighted VOA vial	5 g	5 mL methanol, cool to 4±2 ºC	14 days to analysis
	GRO	SW-846 5035A/8015B SOPs: GR-04-105, GR-03-121	One 10 gram (g) Terracores into pre-preserved tare weighted VOA vial	10 g	10 mL methanol, cool to 4±2 ºC	14 days to Analysis
	DRO	SW-846 3545/3550B/ 8015B SOPs: GR-09-123, GR-09-133, GR-03-122	Two 8-oz wide mouth jar	30g	Cool to 4±2 ºC	14 days to extraction; 40 days to analysis
	PAHs	SW-846 3545/3550B/8270C SIM SOPs: GR-09-133, GR-09-103, GR-04-103		30g	Cool to 4±2 ºC	14 days to extraction; 40 days to analysis
	Pesticides	SW-846 3545/3550B/8081A SOPs: GR-09-133, GR-09-108, GR-03-120		30g	Cool to 4±2 ºC	14 days to extraction; 40 days to analysis
	PCBs	SW-846 3545/3550B/8082 SOPs: GR-09-133, GR-09-108, GR-03-128		30g	Cool to 4±2 ºC	Not applicable
	Metals	SW-846 3050B/6010C/6020A/ 7471A SOPs: GR-01-137, GR-01-139, GR-01-100, GR-01-123, GR-01-129	One 4-oz wide mouth jar	1 g	Cool to 4±2 ºC	180 days to analysis (ICP metals); 28 days (mercury)

1. Refer to the Analytical SOP References table (Worksheet #23).
2. Minimum sample volume or mass requirement.

3. Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted. Per SW-846 Chapter 2, Revision 4, February 2007, no holding time is applicable for PCBs.
4. SVOC analysis is required for groundwater and associated rinsate blanks; PAH analysis is required for surface water and rinsate blanks associated with surface water, soil, and sediment.
5. Refer to Worksheet #15 for analytes to be analyzed by 8270C SIM.
6. Samples collected for dissolved metals analysis will be filtered in the field.

SAP Worksheet #20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates[1]	No. of Laboratory QCs[2]	No. of Field Blanks	No. of Equip. Blanks[3]	No. of VOA Trip Blanks[4]	No. of PT Samples	Total No. of Samples to Lab[5]
Groundwater	VOC	8	1	1	0	1	2	0	12
	SVOCs	8	1	1	0	1	0	0	10
	Pesticides	8	1	1	0	1	0	0	10
	PCBs	8	1	1	0	1	0	0	10
	Total Metals	8	1	1	0	1	0	0	10
	Dissolved Metals[6]	8	1	1	0	1	0	0	10
Soil	VOC	40	4	2	0	2	2	0	48
	PAHs	40	4	2	0	2	0	0	46
	Pesticides	40	4	2	0	2	0	0	46
	PCBs	40	4	2	0	2	0	0	46
	Metals	40	4	2	0	2	0	0	46
	GRO	4	1	1	0	1	1	0	7
	DRO	4	1	1	0	1	0	0	6
Sediment	VOC	7	1	1	0	1	1	0	10
	PAHs	7	1	1	0	1	0	0	9
	Pesticides	7	1	1	0	1	0	0	9
	PCBs	7	1	1	0	1	0	0	9
	Metals	7	1	1	0	1	0	0	9
Surface Water	VOC	7	1	1	0	0	1	0	9
	PAHs	7	1	1	0	0	0	0	8
	Pesticides	7	1	1	0	0	0	0	8
	PCBs	7	1	1	0	0	0	0	8
	Total Metals	7	1	1	0	0	0	0	8
	Dissolved Metals[6]	7	1	1	0	0	0	0	8

1. Collect 1 field duplicate per 10 field samples.
2. Assign 1 Laboratory QC sample per 20 samples for MS/MSD analysis for organics and MS/laboratory duplicate analysis for metals. Collect triple volume of groundwater and surface water for organic analyses and double volume of groundwater for metals analyses. For VOC soil and sediment, triple volume is also required. For other soil and sediment analytical groups, no extra volume is required.

3. Collect 1 equipment blank per 20 field samples except for surface water. (No equipment blank is needed for surface water dissolved metals due to use of disposable tubing and filter).
4. Ship one trip blank per cooler with VOC samples. Ship samples of each matrix in separate coolers.
5. Total number of samples to laboratory does not include the assigned laboratory QC samples.
6. Samples collected for dissolved metals analysis will be filtered in the field.

Following is a description of the QC sample types:

- Field QC Samples

  Field duplicates **will be collected by alternately filling sample containers for a given analytical group from the source being sampled. Field duplicate samples will be shipped blind to the laboratories.**

  Trip blanks **are pre-preserved VOC vials (as described in Worksheet #19) prepared by the laboratory and shipped with the VOC samples.**

  Equipment rinsate blanks **are obtained under representative field conditions by running analyte-free deionized (DI)/distilled water through sample collection equipment after decontamination and prior to use, and placing it in the appropriate sample containers for analysis.**

- **Laboratory QC Samples** - Field samples to be used for laboratory MS/MSD and MS/laboratory duplicate analyses will be assigned by the field sampler on the chain-of-custody form and sample log sheet.

SAP Worksheet #21 -- Project Sampling SOP References Table
(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
CT-04	CT-04 - Sample Nomenclature; Revision 2, March 2009	Tetra Tech	Not applicable	Y	SOP is included in Appendix A. Also see Worksheet # 27 for project-specific details.
CT-05	CT-05 - Database Records and Quality Assurance; Revision 2, January 2001	Tetra Tech	Not applicable	N	SOP is included in Appendix A.
GH-1.2	GH-1.2 - Evaluation of Existing Monitoring Wells and Water Level Measurement; Revision 2, September 2003	Tetra Tech	Electronic water level indicator	Y	SOP is included in Appendix A. Also see Appendix C-1 for project-specific procedures.
GH-1.3	GH-1.3 – Soil and Rock Drilling Methods; Revision 1, June 1999	Tetra Tech	Drilling rig and accessories	N	SOP is included in Appendix A.
GH-1.5	GH-1.5 - Borehole and Sample Logging; Revision 1, June 1999	Tetra Tech	Not applicable	N	SOP is included in Appendix A.
GH-2.5	GH-2.5 – Groundwater Contour Maps and Flow Determinations; Revision 1, February 2009	Tetra Tech	Not applicable	N	SOP is included in Appendix A.
GH-2.8	GH-2.8 – Groundwater Monitoring Well Installation	Tetra Tech	Water quality meter, pump	N	SOP is included in Appendix A. Used only for monitoring well development (subsection 5.4).
HS-1.0	HS-1.0 - Utility Locating and Excavation Clearance; Revision 2, December 2003	Tetra Tech	Magnetometer	N	SOP is included in Appendix A.
SA-1.1	SA-1.1 – Groundwater Sample Acquisition and Onsite Water Quality Testing; Revision 7, April 2008	Tetra Tech	Groundwater quality meters	N	SOP is included in Appendix A.
SA-1.2	SA-1.2 – Surface Water and Sediment Sampling	Tetra Tech	Sampling supplies	N	SOP included in Appendix A.
SA-1.3	SA-1.3 - Soil Sampling; Revision 9, April 2008	Tetra Tech	Sampling supplies, excavator, backhoe	Y	SOP is included in Appendix A. Also see

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
					Appendix C-2 for project-specific procedures.
SA-6.1	SA-6.1 - Non-Radiological Sample Handling ; Revision 3, February 2004	Tetra Tech	Sample Bottle ware, Packaging Material, Shipping Materials	N	SOP is included in Appendix A.
SA-6.3	SA-6.3 - Field Documentation; Revision 3, March 2009	Tetra Tech	Field Logbook, Field Sample Forms, Boring Logs	N	SOP is included in Appendix A.
SA-7.1	SA-7.1 - Decontamination of Field Equipment; Revision 6, January 2009	Tetra Tech	Decontamination Equipment (scrub brushes, phosphate free detergent, de-ionized water)	N	SOP is included in Appendix A.
GW-0001	GW-0001 - Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells; Revision 3, 2010	USEPA Region 1/GW-0001	Submersible pump, multiparameter meter, turbidimeter	N	SOP is included in Appendix A. Also see Appendix C-2 for project-specific procedures.

SAP Worksheet #22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity[1]	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
YSI 600 Series Water Quality Meter	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's Guidance	Operator Correction or Replacement	FOL or designee	GW-0001, Manufacturer's Guidance	None.
Turbidity Meter	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's Guidance; Calibrations must bracket expected values. Initial Calibration Verification (ICV) must be <5 NTU.	Operator Correction or Replacement	FOL or designee	GW-0001, Manufacturer's Guidance	None.
Water Level Indicator and Oil/Water Interface Probe	Visual Inspection Field checks as per manufacturer	Daily Once upon receiving from vendor	0.01 foot accuracy	Operator Correction or Replacement	FOL or designee	GH-1.2	None.
PID	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's Guidance	Operator Correction or Replacement	FOL or designee	Manufacturer's Guidance	To be used to determine the location within a boring interval that is most contaminated for collection of soil sample.

[1] Activities may include: calibration, verification, testing, maintenance.

[2] Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

SAP Worksheet #23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
GR-01-100	Inductively Coupled Plasma Atomic Emission Spectroscopy, 02/05/09 -- Revision 5.7 6010C	Definitive	Water and Solid Metals	Perkin Elmer ICP 3000 3300DV 5300DV	TriMatrix	N
GR-01-123	Mercury by Semi-Automated Cold Vapor Atomic Absorption 02/01/09 -- Revision 5.6, 7470A/7471A	Definitive	Water and Solid Mercury	PSA Millenium Cold Vapor Mercury Analyzer	TriMatrix	N
GR-01-129	Inductively Coupled Plasma-Mass Spectrometry, 03/10/10 -- Revision 3.9 6020A	Definitive	Water and Solid Metals	Perkin Elmer ELAN ICP-MS 6000 6100	TriMatrix	N
GR-01-137	Block Digestion of Solids for ICP and ICPMS, 02/01/09 -- Revision 1.5 3050B	Definitive	Soil Metals	Environmental Express Hot Block	TriMatrix	N
GR-01-139	Block Digestion of Mercury in Soil, Waste, and Oil, 08/15/09 -- Revision 0.2 7471A	Definitive	Soil Mercury	Environmental Express Hot Block	TriMatrix	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
GR-01-140	Digestion of Mercury in Water, Wastewater, and Aqueous Waste, 02/20/09 -- Revision 0.4 7470A	Definitive	Water Mercury	Environmental Express Hot Block	TriMatrix	N
GR-01-147	Block Digestion for Total Metals in Water for ICP, 01/20/09 -- Revision 0.4 3010A	Definitive	Water Metals	Environmental Express Hot Block	TriMatrix	N
GR-01-148	Block Digestion of Aqueous Samples and Extracts for Total/Dissolved Metals for ICPMS, 01/20/09 -- Revision 0.3 3020A	Definitive	Water Metals	Environmental Express Hot Block	TriMatrix	N
GR-03-120	Organochlorine Pesticides Analysis by Gas Chromatography (GC), 02/10/10 -- Revision 4.5, 8081B	Definitive	Pesticides - Aqueous and Solid Analysis	Agilent GC 6890 (ECD-ECD)	Trimatrix	N
GR-03-122	Gasoline Range Organics (GRO) by Gas Chromatography, 08/25/09 -- rev. 3.4, 8015B	Definitive	Water and Soil GRO	Agilent GC 5890 II (PID-FID)	TriMatrix	N
GR-03-123	Diesel Range Organics, 01/20/09 -- rev. 2.3, 8015B	Definitive	Water and Soil DRO	Agilent GC 5890 (FID)	TriMatrix	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
GR-03-128	Polychlorinated Biphenyls by Gas Chromatography (PCBs), 03/19/08 -- Revision 2.4, 8082	Definitive	Water and Solid PCBs	Agilent GC 6890 (ECD-ECD)	TriMatrix	N
GR-04-103	Base/Neutral/Acid Compounds by Gas Chromatography/ Mass Spectrometry, 02/20/09 -- Revision 5.5, 8270C	Definitive	Water and Solid Semi-volatiles/PAHs	Agilent GC 6890 5973N MSD or Agilent GC 6890 5975N MSD	TriMatrix	N
GR-04-104	Volatile Organic Compounds by Purge & Trap Capillary Gas Chromatography/ Mass Spectrometry, 02/20/09 -- Revision 4.5, 8260B	Definitive	Water and Solid Volatiles	Agilent GC 6890 5973N MSD or Agilent GC 7890 5975N MSD	TriMatrix	N
GR-04-105	Closed System Purge & Trap Extraction For Volatile Organic Compounds, 02/20/09 -- Revision 1.2, 5035A	Definitive	Solid Volatiles	EST ENCON P&T Concentrator EST 8100 or Centurion WS P&T Autosampler	TriMatrix	N
GR-09-101	Extraction of Base Neutrals and Acids from Water,	Definitive	Water Semi-volatiles/PAHs	N/A	TriMatrix	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
	03/12/09 -- Revision 3.7, 3510C					
GR-09-103	Extraction of BNA Semi-Volatiles from Soil, Sediment, and Sludge, 03/12/09 -- Revision 3.3, 3550B	Definitive	Soil Semi-volatiles/PAHs	N/A	TriMatrix	N
GR-09-107	Extraction of Organochlorine Pesticides and PCBs from Water, 08/12/09 -- Revision 5.2, 3510C	Definitive	Water PCBs	N/A	TriMatrix	N
GR-09-108	Extraction of Organochlorine Pesticides and PCBs from Soil, Sludge and Wipe Samples, 03/27/09 -- Revision 4.3, 3550B	Definitive	Solid PCBs	N/A	TriMatrix	N
GR-09-123	Diesel Range Organics Extraction (DRO), 10/15/08 -- rev. 0.2, 3550C/8015C	Definitive	Soil DRO	N/A	TriMatrix	N
GR-09-124	Separatory Funnel Liquid-Liquid Extraction of Diesel Range Organics Extraction, 09/10/08 -- rev. 0.1,	Definitive	Water DRO	N/A	TriMatrix	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
	3510C/8015B					
GR-09-133	Pressurized Fluid Extraction, 09/15/07 -- Revision 0.0, 3545	Definitive	Solid DRO/PAH/PCB/Pesticides	N/A	TriMatrix	N
GR-15-100	Sample Receipt and Log-in, 12/31/08 – Revision 3.2	NA	Login and Storage	NA	TriMatrix	N
GR-15-102	Laboratory Waste Disposal, 4/30/09 – Revision 2.1	NA	Waste Disposal	NA	TriMatrix	N
GR-10-104	Chain of Custody, 4/06/09 – Revision 2.2	NA	Storage and Custody	NA	TriMatrix	N

1 Provisions in the analytical SOPs may not match those presented in Worksheets #24 and #28 due to different requirements in the DoD Quality Systems Manual for Environmental Laboratories, (QSM), Version 4.1, for Department of Defense (DoD) projects. The SOPs are not modified for project work. However, TriMatrix has developed analytical checklists for each of the primary methods and a general one for the wet chemistry methods not included in the DoD QSM. The checklists reflect the requirements of the QSM, which are incorporated into Worksheets #24 and #28. TriMatrix provided the checklists to the DoD Environmental Laboratory Accreditation Program (ELAP) accrediting body auditor during the accreditation process.

SAP Worksheet #24 -- Analytical Instrument Calibration Table
(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA (Name or Title)	SOP Reference[1]
GC/MS VOCs	Tuning	Prior to analysis and at the beginning of each 12 hour shift.	See table in SOP. Must meet the ion abundance criteria required by the method. No samples may be accepted without a valid tune.	Determine source of problem by re-tuning using auto or manual tune, clean source, rerun tune.	Analyst/ Supervisor	TriMatrix SOP GR-04-104
	Initial Calibration (ICAL) – A minimum of a 5-point calibration curve is analyzed	Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum 5 point calibration is required.	The percent relative standard deviation (RSD) for each Calibration Check Compound (CCC) must be ≤ 30%. The minimum mean response factor (RF) for each System Performance Check Compound (SPCC) must meet the criteria stated in 8260B. If the RSD for an analyte is > 15%, use a linear curve or quadratic curve (coefficient of determination (r^2) ≥0.99), minimum 6 points for	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA (Name or Title)	SOP Reference[1]
			quadratic quantitation.			
	Initial Calibration Verification (SCV) - 2nd source	Once after each initial calibration, prior to analysis of samples.	Individual compounds must be within ±25% of the true value.	Identify source of problem, correct, repeat calibration, rerun samples	Analyst/ Supervisor	
	Continuing Calibration Verification (CCV)	Analyze a standard at the beginning of each 12 hour shift after a bromofluorobenzene (BFB) tune.	The minimum RF for the SPCCs must meet the criteria stated in 8260B. The CCCs must be ≤ 20 % Difference or %Drift.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
GC/MS SVOCs	Tuning	Prior to analysis and at the beginning of each 12 hour shift.	See table in SOP and/or method. Must meet the ion abundance criteria required by the method. No samples may be accepted without a valid tune.	Determine source of problem by re-tuning using auto or manual tune, clean source, rerun tune.	Analyst/ Supervisor	TriMatrix SOP GR-04-103
	ICAL – A minimum of a 5-point calibration curve is analyzed	Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum 5 point calibration is required.	The %RSD for each CCC must be ≤ 30%. The minimum mean RF for each SPCC must meet the criteria stated in 8270C. If the RSD for an	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA (Name or Title)	SOP Reference[1]
			analyte is > 15%, use a linear curve (r > 0.995) or quadratic curve (r^2 >0.99, minimum 6 points) for quantitation.	affected data.		
	SCV - 2nd source	Once after each initial calibration.	Individual compounds must be within ±25% of true value.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst/ Supervisor	
	CCV	Analyze a standard at the beginning of each 12 hour shift after a Decafluorotriphenyl-phosphine (DFTPP) tune.	The minimum RF for SPCCs must meet the criteria stated in 8270C. The CCCs must be ≤ 20 %D Difference or %Drift.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
GC/ECD (Pesticides/ PCBs)	Breakdown Check (Method 8081A only)	Prior to analysis and at the beginning of each 12 hour shift.	Calculated breakdown for DDT and Endrin must be less than 15%.	Determine the source of the problem, correct and rerun breakdown standard.	Analyst/ Supervisor	TriMatrix SOPs GR-03-120 & GR-03-128
	ICAL – Method 8081A: A minimum of a 5-point calibration curve is analyzed for all analytes.	Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum	Must be ≤20% RSD, or r ≥0.995, or r^2 ≥0.99 with a minimum of 6 points. One point calibration aroclors 1221,	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA (Name or Title)	SOP Reference[1]
	Method 8082: A minimum of a 5-point calibration curve is analyzed for Aroclors 1016/1260 and single point standards for the remaining Aroclors	5 point calibration is required.	1232, 1242, 1248, and 1254 unless present in samples. If present, minimum five point calibration must be performed.	Reanalyze affected data.		
	SCV - 2nd source	Once after each initial calibration.	Individual compounds must be within ±20% of true value.	Identify source of problem, correct, repeat.	Analyst/ Supervisor	
	CCV	Analyze a standard at the beginning and end of the sequence and after every 10 samples.	Individual compounds must be < 20 %D.	If %D > 20% and samples are < PQL, narrate. If %D > 20% only on one column, narrate. Otherwise, reanalyze all samples back to the last acceptable CCV.	Analyst/ Supervisor	
	ICAL –minimum of a 5-point calibration curve is analyzed for all analytes.	Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum 5 point calibration is required.	Must be ≤20% RSD, or r ≥0.995, or r^2 ≥0.99 with a minimum of 6 points. must be performed.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	
	SCV - 2nd source	Once after each	Individual	Identify source of	Analyst/	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA (Name or Title)	SOP Reference[1]
		initial calibration.	compounds must be within ±20% of true value.	problem, correct, repeat.	Supervisor	
	CCV	Analyze a standard at the beginning and end of the sequence and after every 10 samples.	Individual compounds must be < 20 %D.	If %D > 20% and samples are < PQL, narrate. Otherwise, reanalyze all samples back to the last acceptable CCV.	Analyst/ Supervisor	
GC/FID (DRO/GRO)	Retention Time Standards	Prior to analysis of samples	GRO: establish retention time window based on C5 and C12 alkanes DRO: establish retention time window based on C9 and C36 alkanes	Not applicable	Analyst/ Supervisor	TriMatrix SOPs GR-03-121 GR-03-122
	ICAL –minimum of a 5-point calibration curve is analyzed for all analytes.	Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum 5 point calibration is required.	Must be ≤20% RSD, or r ≥0.995, or r^2 ≥0.99 with a minimum of 6 points. must be performed.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	
	SCV - 2nd source	Once after each initial calibration.	Individual compounds must be within ±20% of true value.	Identify source of problem, correct, repeat.	Analyst/ Supervisor	
	CCV	Analyze a standard at the beginning and end of the	Individual compounds must be < 20 %D.	If %D > 20% and samples are < PQL, narrate. Otherwise,	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA (Name or Title)	SOP Reference[1]
		sequence and after every 10 samples.		reanalyze all samples back to the last acceptable CCV.		
ICP-MS Metals	Tuning	Daily prior to ICAL	Mass calibration ≤ 0.1 amu from the true value Resolution <0.9 amu full width at 10% peak height Stability: RSD ≤ 5% for at least four replicate analyses.	Retune instrument then reanalyze tuning solution.	Analyst/ Supervisor	TriMatrix SOP GR-01-129
	Initial Calibration-Minimum of a five point calibration for all target analytes.	Initial Calibration performed daily prior to sample analysis.	The correlation coefficient, r, must be ≥0.995 for all analytes.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
	SCV - 2nd source	Once after each initial calibration, prior to sample analysis.	Individual compounds must be within ±10% of true value.	Identify source of problem, correct, repeat.	Analyst/ Supervisor	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No analytes detected > 2x LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst/ Supervisor	
	CCV	Analyze a standard at the beginning and end of the	Individual compounds must be within 90-110 %R of true value.	Recalibrate and/or perform the necessary equipment	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA (Name or Title)	SOP Reference[1]
		sequence and after every 10 samples.		maintenance. Check the calibration standards. Reanalyze the affected data.		
	Continuing Calibration Blank (CCB)	After every ten samples and at the end of the sequence.	No analytes detected > 2x LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and previous ten samples.	Analyst/ Supervisor	
	Interference Check Standards (ICS - ICSA & ICSB)	At the beginning of each run and after every 20 samples.	ICSA recoveries must be within the absolute value of 2x LOQ for all non-spiked analytes and ICSB recoveries must be within ±20% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
ICP-AES Metals	Initial Calibration-Minimum of a three point calibration for all target analytes.	Initial Calibration performed daily prior to sample analysis.	The correlation coefficient, r, must be ≥0.995 for all analytes.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	Trimatrix SOP GR-01-100
	SCV - 2nd source	Once after each initial calibration,	Individual compounds must	Identify source of problem, correct,	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA (Name or Title)	SOP Reference[1]
		prior to sample analysis.	be within ±10% of true value.	repeat.		
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No analytes detected > 2x LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst/ Supervisor	
	CCV	Analyze a standard at the beginning and end of the sequence and after every 10 samples.	Individual compounds must be within 90-110 %R of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
	Continuing Calibration Blank (CCB)	After every ten samples and at the end of the sequence.	No analytes detected > 2x LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and previous ten samples.	Analyst/ Supervisor	
	Interference Check Standards (ICS - ICSA & ICSB)	At the beginning of each analytical sequence	ICSA recoveries must be within the absolute value of 2x LOD for all non-spiked analytes and ICSB recoveries must be within ±20% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
CVAA (Mercury)	ICAL – A minimum of a 5-	The instrument is calibrated at the	The correlation coefficient, r,	Recalibrate and/or perform	Analyst/ Supervisor	TriMatrix SOP GR-01-123

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA (Name or Title)	SOP Reference[1]
	point calibration curve is analyzed	beginning of each day or if the QC is out of criteria.	must be ≥0.995.	the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.		
	SCV - 2nd source	Once after each initial calibration.	Individual compounds must be within ±10% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data	Analyst/ Supervisor	
	CCV	At the beginning and end of the sequence and after every 10 samples.	Individual compounds must be within 80-120%R of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
	CCB	After every ten samples and at the end of the sequence.	No analytes detected > 2x LOD	Correct the problem, then re-prepare and reanalyze calibration blank and previous ten samples.	Analyst/ Supervisor	

[1] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

[2] Name or title of responsible person may be used.

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person (Name or Title)	SOP Reference[1]
GC/MS Agilent 6890/5973 or 7890/5975	Clean source, replace vacuum pump oil, replace filament, replace inlet liner, replace septa, bake out instrument.	Volatiles and Semivolatiles - Tune instrument	Inspect vacuum pressure.	Clean source and replace vacuum pump oil annually or as needed. Routine maintenance as necessary. Tune instrument after major maintenance. Inspect vacuum pressure daily.	Per instrument manufacturer's instructions.	Inspect system and correct problem; Perform new initial calibration.	GC/MS Analyst	GR-04-103 GR-04-104
EST Encon P&T Concentrator	Clean sparge vessel, replace adsorbent trap, bake out trap.	Soil Volatiles	Check purge flow.	Maintenance as necessary. Purge flow checked weekly.	40ml/minute +/- 2ml/minute	Adjust flow rate or perform leak check. If leak or restriction found fix problem.	GC/MS Analyst	GR-04-104 GR-04-105
GC/ECD/ECD Agilent GC 6890	Replace septa, replace inlet liner, clip column, bake out detectors, recondition column.	Pesticides and PCBs	Check connections, replace disposables, bake out instrument, recondition column and perform leak checks.	Replace liner, septa, and clip column as indicated by instrument change in response and chromatography. Bake out detectors and columns if signal elevated.	Per instrument manufacturer's instructions.	Inspect system; correct problem; perform new initial calibration and affected samples.	GC Analyst	GR-03-120 GR-03-128

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person (Name or Title)	SOP Reference[1]
GC/FID Agilent GC 5890	Replace septa, replace inlet liner, clip column, clean or replace FID jet, bake out detector, recondition column.	TPH-GRO TPH-DRO	Check connections, replace disposables, bake out instrument, recondition column and perform leak checks.	Replace liner, septa, and clip column as indicated by instrument change in response and chromatography. If signal is suppressed, clean or replace FID jet. Bake out detectors and columns if signal elevated.	Per instrument manufacturer's instructions.	Inspect system; correct problem; perform new initial calibration and affected samples.	GC Analyst	GR-03-121 GR-03-122
Perkin Elmer Optima ICP 3000, 3300DV, or 5300DV	Replace pump tubing and clean filters.	Metals	Inspect torch visually, disassemble torch assembly and replace worn or damaged parts.	Replace pump tubing and inspect torch daily. Clean filters weekly. Disassemble torch every 6-8 weeks or as needed.	Per instrument manufacturer's instructions.	Inspect torch assembly and replace any worn or damaged parts. Clean or replace nebulizer and aluminum injector.	Metals Analyst	GR-01-100
Perkin Elmer ELAN ICP/MS 6000 or 6100	Replace pump tubing and filters.	Metals - Tune instrument.	Inspect torch visually, disassemble torch assembly and replace worn or damaged parts.	Perform tune,replace pump tubing and inspect torch daily. Replace filters as needed. Disassemble torch every 6-8 weeks or as needed.	Tune acceptance according to method and instrument manufacturer's instructions.	Inspect torch assembly and replace any worn or damaged parts. Clean or replace torch, nebulizer and aluminum injector.	Metals Analyst	GR-01-129
PSA Millenium	Flush all	Mercury	Check for	Flush tubing	Per instrument	Inspect	Metals Analyst	GR-01-123

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person (Name or Title)	SOP Reference[1]
Mercury Analyzer	tubing, replace pump tubing, replace Perma-pure drying system membrane or entire assembly, replace activated charcoal in exhaust line.		discoloration in Perma-pure drying system.	daily. Change tubing at least weekly. Check Perma-pure system for discoloration monthly. Replace Perma-pure inner membrane every six months. Replace charcoal annually.	manufacturer's instructions.	system and correct problem.		

[1] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

[2] Name or title of responsible person may be used.

SAP Worksheet #26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL/ Tetra Tech
Sample Packaging (Personnel/Organization): FOL/ Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL/ Tetra Tech
Type of Shipment/Carrier: Federal Express
Sample Receipt (Personnel/Organization): TriMatrix Laboratories, Inc. Sample Receiving Group
Sample Custody and Storage (Personnel/Organization): TriMatrix Laboratories, Inc. Sample Receiving Group
Sample Preparation (Personnel/Organization): TriMatrix Laboratories, Inc. Organic Extraction Lab Personnel; TriMatrix Laboratories, Inc. Metals Preparation Lab Personnel.
Sample Determinative Analysis (Personnel/Organization): TriMatrix Laboratories, Inc. Semi-Volatile GC/MS Analysts; Semi-Volatile GC Analysts;. Volatile GC/MS Analysts; Metals Analysts.
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Sample extracts held for minimum 60 days after extraction. Sample digestates held for a minimum of 2 weeks after digestion (This is due to storage limitations. If necessary to reanalyze a sample beyond this, the sample will be redigested).
Biological Sample Storage (No. of days from sample collection): N/A
SAMPLE DISPOSAL
Personnel/Organization: TriMatrix Laboratories, Inc. Sample Receiving Group

SAP Worksheet #27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

Sample Designation and Tracking System

Each sample collected will be assigned a unique sample tracking number used to catalog the results. The sample tracking number will consist of alpha-numeric characters identifying the site, sample medium, location, and depth or date. Any other pertinent information regarding sample identification will be recorded on the sample log sheets or in the field logbooks. The alpha-numeric (A-N) coding to be used in the sample system is described below.

AAA	-	AA	-	AANN	-	(NNNN)
(Site ID)	-	(Medium)	-	(Location)	-	(Depth or Date)

Site identifier: CRF

Medium identifier: "GW" for groundwater samples, "SS" for surficial soil samples, "SO" for subsurface soil samples, "SD" for sediment samples, "SW" for surface water samples.

Sample location identifier: Each sample station will be assigned a test pit, well, soil boring, surface water, or sediment identification as a unique location identifier.

Depth/Date: For soil and sediment samples, this portion of the tracking number will represent the depth in feet bgs (or for sediment, in inches below sediment surface) from which the sample was collected; e.g., for soil samples collected from the 12 to 14 feet bgs depth interval, this portion of the sampling tracking number will be "1214"; for sediment collected from 0 to 6 inches, this portion of the sample tracking number will be "0006". For groundwater and surface water samples, this portion of the sample tracking number will represent the date of the sample collection, "MMYY".

For example: A groundwater sample from well MW01 collected in September 2010 and sent for laboratory analysis will be identified as: CRF-GW-MW01-0910. A soil sample collected from a depth of 8 to 10 feet bgs at location SB01 will be identified as: CRF-SO-SB01-0810.

Quality Control Samples QC samples will use the same coding system as for the environmental samples. Field QC designations will conform to the following formats:

- Field Duplicates: Blind field duplicate samples will be designated such that the location designation will be replaced with "DUP" followed by a sequential value (the n'th duplicate sample collected during that sampling event) and the date (MMDDYY). The sample log sheet will note from which sample location the duplicate was collected. For example, the first groundwater field

duplicate sample collected during an event that was collected on September 4, 2010 would be labeled CRF-GW-DUP01-090410 and the fourth field duplicate collected during that event, collected on September 12, 2010, would be CRF-GW-DUP04-091210.

- Trip Blanks: Trip blank sample identifiers will consist of the site ID, the medium of the samples associated with the trip blank, the label "-TB", a sequential value (the n'th trip blank collected for that medium during that sampling event) and the date (MMDDYY). For example, the first trip blank for the sampling event to be shipped with groundwater samples on September 4, 2010 would be labeled CRF-GW-TB01-090410.

- Rinsate Blanks: Rinsate blank sample identifiers will consist of the site ID, the medium of the samples associated with that piece of equipment, the label "-RB", a sequential value (the n'th rinsate blank collected for that medium during that sampling event), and the date (MMDDYY). Example: CRF-SO-RB01-092810.

- Laboratory QC samples Matrix spike and matrix spike duplicate samples have no separate sample identifier codes, but are noted on the chain-of-custody record and sample log sheet.

Sample Collection Documentation

A project-specific field logbook will be used to keep daily records of significant events, observations, and measurements during field investigations. The field logbook also will be used to document all sampling activities. Logbook entries will be made with indelible ink to provide a permanent record and any errors found in the logbook will be verified, crossed-through with a single line, and initialed by the person discovering the error. Field logbooks will be maintained according to Tetra Tech SOP SA-6.3 (Appendix A).

Field sample log sheets and monitoring well purge data sheets will be used to document sample collection details, while other observations and activities will be recorded in the field logbook. Examples of the field documentation forms can be found in Appendix B.

Field Sample Handling and Chain-of-Custody Procedures

Custody of samples must be maintained and documented at all times to ensure the integrity of a sample from collection through analysis. An accurate written record is necessary to trace the possession and handling of the sample, this documentation is referred to as the "chain-of-custody" form. Chain-of-custody begins when samples are collected in the field, and is maintained by storing the samples in secure areas until custody can be passed on. All samples will be delivered to the laboratory

accompanied by a chain-of-custody form that will describe the sample identifiers, the analytical parameters, and the persons who are responsible for the sample integrity.

Prior to sample collection, sample containers will be labeled with the sample location number, sampler's name, date, sample preservation, and analytical fraction.

Following collection, samples will be placed on ice in a secure cooler and attended by Tetra Tech personnel or placed in locked vehicles or designated storage areas until analysis or shipment to an off-site laboratory. Chain-of-custody procedures are described in further detail in the following Tetra Tech SOPs: SA-6.3, Field Documentation and SA-6.1, Non-Radiological Sample Handling (Appendix A).

The samples will be shipped to the laboratories in coolers packed with ice and bubble wrap or equivalent packing material to cushion the samples to prevent breakage and to maintain the required temperature for the samples. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory upon sample receipt to verify acceptable sample preservation temperature. The coolers will be taped and sealed with a signed custody seal to ensure the chain-of-custody is maintained. Samples will be shipped to the laboratory by an overnight courier to ensure that maximum sample holding times are not exceeded. The maximum allowable sample holding times for all analyses are presented in Worksheet #19. This worksheet also lists the sample containers, chemical preservatives, and temperature condition requirements to maintain the integrity of the samples for each analysis and medium.

Each sample collected will be assigned a unique sampling tracking number, as described above. The sample number, sample collection date and time, person collecting the sample and a list of the sample analyses to be performed will be recorded on each container, and also on the chain-of-custody form. The chain-of-custody form is a two-part form: the original accompanies the samples to the analytical laboratory, and the copy will be archived in the project files. The following information will be recorded on the chain-of-custody form:

- Project name and number
- Sample matrix
- Sample collector's name
- Dates/times of sample collection
- Sample identification numbers
- Number and type of containers for each sample aliquot
- Type of preservation
- Quality control (QC) sample designation

- Analysis method
- Special handling instructions
- Destination of samples
- Name, date, time, and signature of each individual releasing the shipping container

The field crew will attempt to identify any potentially high-concentration samples (based on the field screening results or other observations) on the chain-of-custody form.

Laboratory Custody Procedures

The TriMatrix Laboratories sample receipt and chain-of-custody procedure is detailed in TriMatrix Laboratories' SOP GR-15-100 and GR-15-102 (Appendix E). The laboratory sample custodian will inspect the integrity of the cooler custody seals and measure the temperature of the samples received using the "Temperature Blank" container included in each cooler. The samples will be checked against the chain-of-custody form for holding times, sample identification, and integrity. The samples will be logged into the laboratory management system. Custody of the samples will be maintained and recorded in the laboratory from receipt to analysis and this record will be included with the data package deliverables.

TriMatrix Laboratories SOP GR-15-102 Rev 2.1 (Appendix E) describes the laboratory procedures for disposal of the environmental samples.

SAP Worksheet #28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil, Sediment Groundwater, Surface Water
Analytical Group	VOCs
Analytical Method/ SOP Reference	SW-846 8260B TriMatrix SOP GR-04-104

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or less.	No target analytes > ½ LOQ (> LOQ for common laboratory contaminants) unless target analytes in field samples are > 10x those in method blank.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results	GC/MS Analyst	Accuracy/bias-Contamination	No target analytes > ½ LOQ (> LOQ for common laboratory contaminants) unless target analytes in field samples are > 10x those in method blank.
Surrogates	Four per sample: Dibromofluoromethane 1,2-Dichloroethane Toluene-d8 4-Bromofluorobenzene	Table G-3 DOD QSM for %R. See attached Table 28-1 in Appendix E.	Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	GC/MS Analyst	Accuracy/bias	DOD QSM limits for %R. See attached Table 28-1.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per SDG or every 20 samples.	Tables G-4 and G-5 DoD QSM Version 4.1 See attached Table 28-1.	Check for errors in calculations and spike preparation. Check unspiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	GC/MS Analyst	Precision / Accuracy / Bias	DOD QSM limits for %R. MS/MSD RPD ≤ 30%. See attached Table 28-1.
Laboratory Control Sample (LCS)	One per batch of 20 or less.	Tables G-4 and G-5 DoD QSM Version 4.1 See attached Table 28-1.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	GC/MS Analyst	Accuracy / Bias	DOD QSM limits for %R. See attached Table 28-1.

Matrix	Soil, Sediment Groundwater, Surface Water
Analytical Group	VOCs
Analytical Method/ SOP Reference	SW-846 8260B TriMatrix SOP GR-04-104

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample Duplicate (LCSD) (Not Required)	Used if insufficient sample supplied for batch MS/MSD.	Table G-4 Department of Defense (DoD) Quality Systems Manual (QSM) Version 4.1 See attached Table 28-1.		GC/MS Analyst	Precision / Accuracy / Bias	DOD QSM limits for %R. LCS/LCSD RPD ≤ 30%. See attached Table 28-1.
Internal Standards (IS)	Three per sample: Fluorobenzene Chlorobenzene-d5 1,4-Dichlorobenene-d5	Retention time +/- 30 seconds from retention time (RT) of the midpoint calibration initial calibration standard, and the Extracted Ion Current Profile (EICP) area within -50% to +100 % of initial calibration midpoint standard.	Inspect instrument for malfunctions. Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	GC/MS Analyst	Accuracy / Bias	Retention time +/- 30 seconds from retention time (RT) of the midpoint calibration initial calibration standard, and EICP area within -50% to +100 % of initial calibration midpoint standard.

Matrix	Soil, Sediment, Groundwater, Surface Water
Analytical Group	SVOCs, PAHs
Analytical Method/ SOP Reference	SW846 8270C/8270C SIM TriMatrix SOP GR-04-103

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or less.	No target analytes > ½ LOQ (> LOQ for common laboratory contaminants) unless target analytes in field samples are > 10x those in method blank.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results	GC/MS Analyst	Accuracy/bias-Contamination	No target analytes > ½ LOQ (> LOQ for common laboratory contaminants) unless target analytes in field samples are > 10x those in method blank.
Surrogates	Six per sample: 2-Fluorophenol Phenol-d6 2,4,6-Tribromophenol Nitrobenzene-d5 2-Fluorobiphenyl ortho-Terphenyl	Tables G-2 and G-3 DOD QSM for %R. See attached Table 28-1.	Check for possible matrix effects. If none found, re-extract and reanalyze affected sample if sufficient sample is available. Qualify data as needed.	GC/MS Analyst	Accuracy/bias	DOD QSM limits for %R. See attached Table 28-1.
MS/MSD	One per SDG or every 20 samples.	Tables G-2, G-6, and G-7 DoD QSM Version 4.1 See attached Table 28-1.	Check for errors in calculations and spike preparation. Check unspiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	GC/MS Analyst	Precision / Accuracy / Bias	DOD QSM limits for %R. MS/MSD RPD ≤ 30%. See attached Table 28-1.
LCS	One per batch of 20 or less.	Tables G-2, G-6, and G-7 DoD QSM Version 4.1 See attached Table 28-1.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	GC/MS Analyst	Accuracy/bias	DOD QSM limits for %R. See attached Table 28-1.
LCSD (Not Required)	Used if insufficient sample supplied for batch MS/MSD.	Tables G-2, G-6, and G-7 DoD QSM Version 4.1 See attached Table 28-1.	Qualify failing analytes as estimated.	GC/MS Analyst	Precision / Accuracy / Bias	DOD QSM limits for %R. LCS/LCSD RPD ≤ 30%. See attached Table 28-1.

Matrix	Soil, Sediment, Groundwater, Surface Water
Analytical Group	SVOCs, PAHs
Analytical Method/ SOP Reference	SW846 8270C/8270C SIM TriMatrix SOP GR-04-103

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards	Six per sample: 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Retention time +/- 30 seconds from RT of the midpoint calibration initial calibration standard, and the EICP area within -50% to +100 % of initial calibration midpoint standard.	Inspect instrument for malfunctions. Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	GC/MS Analyst	Accuracy / Bias	Retention time +/- 30 seconds from RT of the midpoint calibration initial calibration standard, and the EICP area within -50% to +100 % of initial calibration midpoint standard.

Matrix	Soil, Sediment, Groundwater, Surface Water
Analytical Group	DRO
Analytical Method/ SOP Reference	SW846 8015B TriMatrix SOP GR-03-122

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or less.	No target analytes > ½ LOQ unless target analytes in field samples are > 10x those in method blank.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results	GC Analyst	Accuracy/bias-Contamination	No target analytes > ½ LOQ unless target analytes in field samples are > 10x those in method blank.
Surrogates	1 per sample ortho-terphenyl	% Recovery: Waters 45-136% Soils 44-137%	Check for possible matrix effects. If none found, re-extract and reanalyze affected sample if sufficient sample is available. Qualify data as needed.	GC Analyst	Accuracy/bias	% Recovery: Waters 45-136% Soils 44-137%
MS/MSD	One per batch of 20 or less.	% Recovery: 50-150% MS/MSD RPD ≤ 30%	Check for errors in calculations and spike preparation. Check unspiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated	GC Analyst	Accuracy/bias & Precision	% Recovery: 50-150% MS/MSD RPD ≤ 30%
LCS	One per batch of 20 or less.	% Recovery: 50-150%	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	GC Analyst	Accuracy/bias	% Recovery: 50-150%
LCS Duplicate	One per batch of 20 or less.	% Recovery: 50-150% MS/MSD RPD ≤ 30%	Qualify failing analytes as estimated.	GC Analyst	Precision / Accuracy / Bias	% Recovery: 50-150% MS/MSD RPD ≤ 30%

Matrix	Soil, Sediment, Groundwater, Surface Water
Analytical Group	GRO
Analytical Method/ SOP Reference	SW846 8015B TriMatrix SOP GR-03-121

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or less.	No target analytes > ½ LOQ unless target analytes in field samples are > 10x those in method blank.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results	GC Analyst	Accuracy/bias-Contamination	No target analytes > ½ LOQ unless target analytes in field samples are > 10x those in method blank.
Surrogates	1 per sample ortho-terphenyl	% Recovery: Waters 86-113% Soils 76-113%	Check for possible matrix effects. If none found, re-extract and reanalyze affected sample if sufficient sample is available. Qualify data as needed.	GC Analyst	Accuracy/bias	% Recovery: Waters 86-113% Soils 76-113%
MS/MSD	One per batch of 20 or less.	% Recovery: Waters 75-122% Soils 72-118% MS/MSD RPD ≤ 30%	Check for errors in calculations and spike preparation. Check unspiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated	GC Analyst	Accuracy/bias & Precision	% Recovery: Waters 75-122% Soils 72-118% MS/MSD RPD ≤ 30%
LCS	One per batch of 20 or less.	% Recovery: Waters 75-122% Soils 72-118%	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	GC Analyst	Accuracy/bias	% Recovery: Waters 75-122% Soils 72-118%
LCS Duplicate	One per batch of 20 or less.	% Recovery: Waters 75-122% Soils 72-118% MS/MSD RPD ≤ 30%	Qualify failing analytes as estimated.	GC Analyst	Precision / Accuracy / Bias	% Recovery: Waters 75-122% Soils 72-118% MS/MSD RPD ≤ 30%

Matrix	Soil, Sediment, Groundwater, Surface Water
Analytical Group	Pesticides/PCBs
Analytical Method/ SOP Reference	SW846 8081/8082 TriMatrix SOPs GR-03-120, GR-03-128

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or less.	No target analytes > ½ LOQ unless target analytes in field samples are > 10x those in method blank.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results	GC Analyst	Accuracy/bias-Contamination	No target analytes > ½ LOQ unless target analytes in field samples are > 10x those in method blank.
Surrogates	Two per sample tetrachloro-m-xylene and decachlorobiphenyl	Table G-3 DOD QSM for %R. See attached Table 28-1.	Check for possible matrix effects. If none found, re-extract and reanalyze affected sample if sufficient sample is available. Qualify data as needed.	GC Analyst	Accuracy/bias	DOD QSM limits for %R. See attached Table 28-1.
MS/MSD	One per SDG or every 20 samples.	Pesticides: Tables G-14 and G-15 DoD QSM Version 4.1 PCBs: Tables G16 and G-17 DoD QSM Version 4.1 See attached Table 28-1.	Check for errors in calculations and spike preparation. Check unspiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	GC Analyst	Precision / Accuracy / Bias	DOD QSM limits for %R. MS/MSD RPD ≤ 30%. See attached Table 28-1.
LCS	One per batch of 20 or less.	Pesticides: Tables G-14 and G-15 DoD QSM Version 4.1 PCBs: Tables G16 and G-17 DoD QSM Version 4.1 See attached Table 28-1.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	GC Analyst	Accuracy/bias	DOD QSM limits for %R. See attached Table 28-1.

Matrix	Soil, Sediment, Groundwater, Surface Water
Analytical Group	Pesticides/PCBs
Analytical Method/ SOP Reference	SW846 8081/8082 TriMatrix SOPs GR-03-120, GR-03-128

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCSD (Not Required)	Used if insufficient sample supplied for batch MS/MSD.	Pesticides: Tables G-14 and G-15 DoD QSM Version 4.1 PCBs: Tables G16 and G-17 DoD QSM Version 4.1 See attached Table 28-1.	Qualify failing analytes as estimated.	GC Analyst	Precision / Accuracy / Bias	DOD QSM limits for %R. LCS/LCSD RPD ≤ 30%. See attached Table 28-1.
Second Column Confirmation	Used to confirm hit on primary column.	RPD ≤40%.	Report the higher of the two values unless obvious matrix interference exists.	GC Analyst	Confirmation	RPD ≤40%.

Matrix	Soil, Sediment, Groundwater, Surface Water
Analytical Group	ICP Metals (ICP-AES) and Mercury
Analytical Method/ SOP Reference	SW846 6010B/7470A/7471A TriMatrix SOPs GR-01-100 and GR-01-123

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples	No target analytes > ½ LOQ (> LOQ for common laboratory contaminants) unless target analytes in field samples are > 10x those in method blank.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results	Metals Analyst	Accuracy/bias-Contamination	No target analytes > ½ LOQ (> LOQ for common laboratory contaminants) unless target analytes in field samples are > 10x those in method blank.
Interference Check Sample (ICP only)	At the beginning of each analytical sequence	ICSA: Recoveries must be within the absolute value of 2x LOD for all non-spiked analytes ICSB: Recoveries must be within ±20% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Metals Analyst	Accuracy/bias	ICSA: Recoveries must be within the absolute value of 2x LOD for all non-spiked analytes ICSB: Recoveries must be within ±20% of true value.

Matrix	Soil, Sediment, Groundwater, Surface Water
Analytical Group	Pesticides/PCBs
Analytical Method/ SOP Reference	SW846 8081/8082 TriMatrix SOPs GR-03-120, GR-03-128

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	One per digestion batch of 20 or fewer samples.	Table G-18 DoD QSM Version 4.1 See attached Table 28-1	Check for errors in calculations and spike preparation. Check unspiked sample results for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Metals Analyst	Precision / Accuracy / Bias	DoD QSM limits for %R, if sample < 4x spike added. RPD ≤20. See attached Table 28.1
Post-Digestion Spike (ICP only)	One per digestion batch of 20 or fewer samples.	Recovery within 75-125% of expected value	Apply J-flag to associated sample result. Not necessary if analyte concentration < 50 x MDL.	Metals Analyst	Accuracy / Bias	DoD QSM limits of 75-125 %R.
LCS	One per digestion batch of 20 or fewer samples.	Table G-18 DoD QSM Version 4.1. See attached Table 28-1	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Metals Analyst	Accuracy / Bias	DoD QSM limits for %R. See attached Table 28.1
Serial Dilution (ICP only)	One per digestion batch.	If original sample result is at least 50x the LOQ, 5-fold dilution must be ≤10% RPD.	Analyze Post Digestion Spike and narrate the analytes that fail criteria.	Metals Analyst	Precision / Accuracy / Bias	If original sample result is at least 50x the LOQ, 5-fold dilution must be ≤10% RPD.

Matrix	Soil, Sediment, Groundwater, Surface Water
Analytical Group	ICP Metals (ICP-MS)
Analytical Method/ SOP Reference	SW846 6020 TriMatrix SOP GR-01-129

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples	No target analytes > ½ LOQ (> LOQ for common laboratory contaminants) unless target analytes in field samples are > 10x those in method blank.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results	Metals Analyst	Accuracy/bias-Contamination	No target analytes > ½ LOQ (> LOQ for common laboratory contaminants) unless target analytes in field samples are > 10x those in method blank.
Interference Check Sample	At the beginning of each analytical sequence	ICSA: Recoveries must be within the absolute value of 2x LOD for all non-spiked analytes ICSB: Recoveries must be within ±20% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Metals Analyst	Accuracy/bias	ICSA: Recoveries must be within the absolute value of 2x LOD for all non-spiked analytes ICSB: Recoveries must be within ±20% of true value.
MS/MSD	One per digestion batch of 20 or fewer samples.	Table G-18 DoD QSM Version 4.1 See attached Table 28-1	Check for errors in calculations and spike preparation. Check unspiked sample results for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Metals Analyst	Precision / Accuracy / Bias	DoD QSM limits for %R, if sample < 4x spike added. RPD ≤20. See attached Table 28.1
Post-Digestion Spike	One per digestion batch of 20 or fewer samples.	Recovery within 75-125% of expected value	Apply J-flag to associated sample result. Not necessary if analyte concentration < 50 x MDL.	Metals Analyst	Accuracy / Bias	DoD QSM limits of 75-125 %R.
LCS	One per digestion batch of 20 or fewer samples.	Table G-18 DoD QSM Version 4.1. See attached Table 28-1	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Metals Analyst	Accuracy / Bias	DoD QSM limits for %R. See attached Table 28.1
Serial Dilution	One per digestion batch.	If original sample result is at least 50x the LOQ, 5-fold dilution must be ≤10% RPD.	Analyze Post Digestion Spike and narrate the analytes that fail criteria.	Metals Analyst	Precision / Accuracy / Bias	If original sample result is at least 50x the LOQ, 5-fold dilution must be ≤10% RPD.

Matrix	Soil, Sediment, Groundwater, Surface Water
Analytical Group	ICP Metals (ICP-MS)
Analytical Method/ SOP Reference	SW846 6020 TriMatrix SOP GR-01-129

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standard	Seven per sample: Bismuth Germanium Holmium Indium Lithium(+6) Rhodium Terbium	IS intensity within 60-120% of the intensity of the IS in the ICAL	Reanalyze sample at 5-fold dilution.	Metals Analyst	Accuracy	IS intensity within 60-120% of the intensity of the IS in the ICAL

Note: Table 28-1 is included in Appendix E.

Analysis/ Method:	GRO/DRO/EPA SW-846 Method 8015B					
Laboratory QC Sample:	**Frequency /Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator**	**Measurement Performance Criteria**
Temperature Blank	1 per cooler	4 ± 2 °C	Notify TtNUS Project Chemist or PM who will address the impact to the project and determine the appropriate action	Laboratory Sample Receiver, TtNUS Chemist or PM, Data Validator	Precision / Representativity / Bias	4 ± 2 °C
Method Blank	One per batch of 20 or less, prior to sample analysis	No target compounds ≥ ½ PQL except common lab contaminants which should be < RL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target analytes > ½ PQL (> PQL for common laboratory contaminants), unless target analytes in field samples are > 10x those in method blank.
Surrogates	1 per sample	Statistically derived limits.	(1) Reprepare and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Statistically derived limits.
Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)	One per batch of 20 or less	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits.	(1) Evaluate and reanalyze if possible (2) If an MS/MSD was performed in the same 12 hour clock and acceptable, narrate. (3) If the LCS recoveries are high but the sample results are <QL, narrate otherwise reprepare and reanalyze	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits.

SAP Worksheet #29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Sample Collection Documents and Records: • Field logbook (and sampling notes) • Field sample forms • Chain-of-custody records • Sample shipment airbills • Equipment calibration logs • Photographs • FTMR forms • SAP • Field Sampling SOPs	Tetra Tech project file; results will be provided in the Investigation Report.
Laboratory Documents and Records: • Sample receipt/login form • Sample storage records • Sample preparation logs • Standard traceability logs • Equipment calibration logs • Sample analysis run logs • Equipment maintenance, testing, and inspection logs • Corrective Actions forms • Reported results for standards, QC checks, and QC samples • Data completeness checklists • Sample storage and disposal records • Telephone logs • Extraction/clean-up records • Raw data	Tetra Tech project file; long-term data package storage at third party commercial document storage firm.
Data Assessment Documents and Records: • Field Sampling Audit Checklist (if an audit is conducted) • Data validation Memoranda • Investigation Report	Tetra Tech project file.

SAP Worksheet #30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Soil, Sediment, Groundwater, Surface Water	VOCs	See Worksheet #18	SW-846 8260B	21 days	TriMatrix Laboratories 5560 Corporate Exchange Court Grand Rapids, Michigan 49512 Contact: Walt Roudebush (Project Manager) Phone: 616-975-4561 E-mail: roudebushw@trimatrixlabs.com	Not Applicable
	GRO	See Worksheet #18	SW-846 8015B			
	Pesticides	See Worksheet #18	SW-846 8081A			
	PCBs	See Worksheet #18	SW-846 8082			
	Metals (Total)	See Worksheet #18	SW-846 6010C/6020A/ 7470A/7471A			
	DRO	See Worksheet #18	SW-846 8015B			
Groundwater, Surface Water	Dissolved Metals	See Worksheet #18	SW-846 6010C/6020A/ 7470A			
Groundwater	SVOCs	See Worksheet #18	SW-846 8270C/ 8270C SIM			
Soil, Sediment, Surface Water	PAHs	See Worksheet #18	SW-846 8270C SIM			

SAP Worksheet #31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Laboratory System Audit[1]	Every 24 months	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body	Trimatrix QA Manager or Laboratory Manager	Trimatrix QAM or Laboratory Manager	DoD ELAP Accrediting Body

[1] Trimatrix has successfully completed the DoD's Environmental Laboratory Accreditation Program (ELAP) audit. Copies of Trimatrix's DoD ELAP accreditation certificates are included in Appendix E.

SAP Worksheet #32 -- Assessment Findings and Corrective Action Responses

(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Field Sampling System Audit (1)	Audit checklist (as per Navy Installation Restoration Chemical Data Quality Manuel [IRCDQM]) and written audit report	Tom Campbell, PM, Tetra Tech; Mike Horton, FOL, Tetra Tech; John Trepanowski Program Manager; Garth Glenn, Deputy Program Manager, Tetra Tech	Dependent on the finding; if major a stop work may be issued immediately; however, if minor within 1 week of audit	Written memo	Tom Campbell, PM, Tetra Tech; Mike Horton, FOL, Tetra Tech; John Trepanowski Program Manager; Garth Glenn Perry, Deputy Program Manager, Tetra Tech	Within 48 hours of notification
Laboratory System Audit	Written audit report	Rick Wilburn, Trimatrix Lab QAM,	Not specified by DoD ELAP	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP

1 Audits are scheduled at the Tetra Tech program level and may or may not include this project.

SAP Worksheet #33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation Report	Per sample delivery group (SDG)	Once validation is complete	Tetra Tech DVM or designee	Tetra Tech PM, project file
Major Analysis Problem Identification (internal memo)	When persistent analysis problems are detected	Immediately	Tetra Tech CLEAN QAM	Tetra Tech PM, CLEAN QAM, Program Manager, and project file
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately	Trimatrix QAM	Tetra Tech PM, project file

SAP Worksheet #34 -- Verification (Step I) Process Table

(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign each Chain-of-Custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The Chain-of-Custody forms will be signed by the sampler and a copy will be retained for the project file, the PM, and the data validator. See SOP SA-6.3.	Internal	Field Crew, Tetra Tech
	The laboratory Sample Custodian will review the sample shipment for completeness and integrity and will sign accepting the shipment. The laboratory Sample Custodian will check that the Chain-of-Custody form was signed and dated by the Tetra Tech FOL or designee relinquishing the samples.	External	Laboratory Sample Custodian, Trimatrix Laboratories
SAP Sample Tables	Planned samples listed in the SAP will be verified to have been collected.	Internal	FOL and Field Crew, Tetra Tech
Sample Log Sheets	Log sheets that are completed as samples are collected in the field will be verified for completeness and will be maintained in the Tetra Tech project file.	Internal	PM (Tom Campbell), FOL, or designee, Tetra Tech
Sample Coordinates	Sample locations will be verified to be correct and in accordance with the SAP (compare map of proposed locations to map of actual locations).	Internal	PM, FOL, or designee, Tetra Tech
Field QC Samples	Verify that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory QAM will sign the case narrative for each data package.	Internal	Laboratory QAM, Trimatrix Laboratories
	Verify that the data package contains all the elements required by the functional guidelines and the scope of work. This occurs as part of the data validation process.	External	Data Validator, Tetra Tech
Electronic Data Deliverables	The electronic data will be compared to the Chain-of-Custody form and hard copy data package to verify accuracy and completeness.	External	Data Validator, Tetra Tech

SAP Worksheet #35 -- Validation (Steps IIa and IIb) Process Table

(UFP-QAPP Manual Section 5.2.2) (Figure 37 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa/IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Chain-of-Custody Forms	Ensure that the custody and integrity of the samples were maintained from collection to analysis and that custody records are complete and any deviations are recorded.	Data Validator, Tetra Tech
IIa	Holding Times	Verify that the samples were shipped and stored at the required temperature and that the sample pH values for chemically preserved samples meet the requirements listed in Worksheet #19. Verify that the analyses were performed within the holding times listed in Worksheet #19.	Data Validator, Tetra Tech
IIa	Data Results	Verify that the summary form results match the raw data.	Data Validator, Tetra Tech
IIa/IIb	Laboratory Data Results	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Verify that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Data Validator, Tetra Tech
IIa/IIb	Field and Laboratory Duplicate Analyses for Precision	Verify the field sampling precision by checking the RPD for field duplicate samples. Verify laboratory precision by checking the RPDs from MS/MSD analyses. Ensure compliance with the methods and project MPC accuracy goals listed in Worksheets #12 and #28.	Data Validator, Tetra Tech
IIa/IIb	Sample Results for Representativeness	Verify that the laboratory recorded the temperature of each sample at sample receipt and the pH of chemically preserved samples to ensure sample integrity from sample collection to analysis.	Data Validator, Tetra Tech
IIa/IIb	Project Action Levels	Discuss the impact of matrix interferences or sample dilutions performed, because of the high concentration of one or more contaminants, on the other target compounds reported as not detected. Document this usability issue and inform the PM.	Data Validator, Tetra Tech
	Project Action Levels	Review and add PALs to the laboratory electronic data deliverable. Flag samples and notify the PM of samples that exceed the PALs as listed in Worksheet #15.	PM or designee, Tetra Tech
IIa/IIb	Data Validation Report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all data qualifications. Print a copy of the project database, qualified data depicting data qualifiers, and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	Data Validator, Tetra Tech

Step IIa/IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa, IIb	SAP QC Sample Documentation	Verify that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Verify that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the laboratory shall have contacted the Tetra Tech Project Chemist or PM.	Data Validator, Tetra Tech
IIa, IIb	Documentation of Analytical Reports for Completeness	Ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Verify all data have been transferred correctly and completely to the final SQL database.	Data Validator, Tetra Tech
IIb	Project Quantitation Limits for Sensitivity	Verify that the QLs listed in Worksheet #15 were achieved.	Data Validator, Tetra Tech
IIb	Analytical Data Deviations	Determine the impact of any deviation from sampling or analytical methods, SOP requirements, and matrix interferences on the analytical results.	Data Validator, Tetra Tech

SAP Worksheet #36 –Analytical Data Validation (Steps IIa and IIb) Summary Table

(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Groundwater, Soil, Sediment, Surface Water	VOCs, SVOCs, PAHs, GRO/DRO	Tier II[1] data validation. Project-specific criteria for VOCs by SW-846 8260B, SVOCs and PAHs by SW-846 8270C/8270C SIM, GRO/DRO by SW-846 8015B are listed in Worksheets #12, #15, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (USEPA, 1996) will be applied using these criteria.	Tetra Tech Project Chemist or Data Validators
IIa and IIb	Groundwater, Soil, Sediment, Surface Water	Pesticides, PCBs	Tier II[1] data validation. Project-specific criteria for pesticides by SW-846 8081A and PCBs by SW-846 8082 are listed in Worksheets #12, #15, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, February 2004 (USEPA, 2004) will be applied using these criteria.	Tetra Tech Project Chemist or Data Validators
IIa and IIb	Groundwater, Soil, Sediment, Surface Water	Metals	Tier II[1] data validation. Project-specific criteria for metals by SW-846 6010B are listed in Worksheets #12, #15, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, November 2008 (USEPA, 2008) will be applied using these criteria.	Tetra Tech Project Chemist or Data Validators

1. As specified in the USEPA Region 1 Tiered Organic and Inorganic Data Validation Guidelines (July 1, 1993).

SAP Worksheet #37 -- Usability Assessment
(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment

The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics:

Completeness

- For each matrix that was scheduled to be sampled, the FOL acting on behalf of the project team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified the Tetra Tech PM and risk assessor will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech PM will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Precision

- The Project Chemist acting on behalf of the project team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets 12 and 28. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

Accuracy

- The Project Chemist acting on behalf of the project team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet 28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, matrix spike, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness

- A project scientist identified by the Tetra Tech PM and acting on behalf of the project team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with the SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

Comparability

- The Project Chemist acting on behalf of the project team will determine whether the data generated under this project are sufficiently comparable to historical site data generated by different methods and for samples collected using different procedures and under different site conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the Project Chemist indicates that such quantitative analysis is required.

Sensitivity

- The Project Chemist acting on behalf of the project team will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets

for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Project Chemist will enlist the help of the project risk assessor to evaluate deviations from planned sensitivity goals.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The project team members identified by the project manager will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight of evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to one-half the LOD. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate samples will be used to represent the concentration at a particular sampled location.

Identify the personnel responsible for performing the usability assessment:

The Tetra Tech PM, Project Chemist, FOL, and Project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM, the EPA Remedial Project Manager, and the State of Rhode Island Project Manager. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

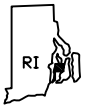
Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary.

FIGURES

BASE MAP IS A PORTION OF THE FOLLOWING 7.5 X 15 MINUTE U.S.G.S. QUADRANGLE:
PRUDENCE ISLAND, RHODE ISLAND, 1955, PHOTOREVISED 1970 AND 1975

GRAPHIC SCALE
0 0.5 MILE 1 MILE

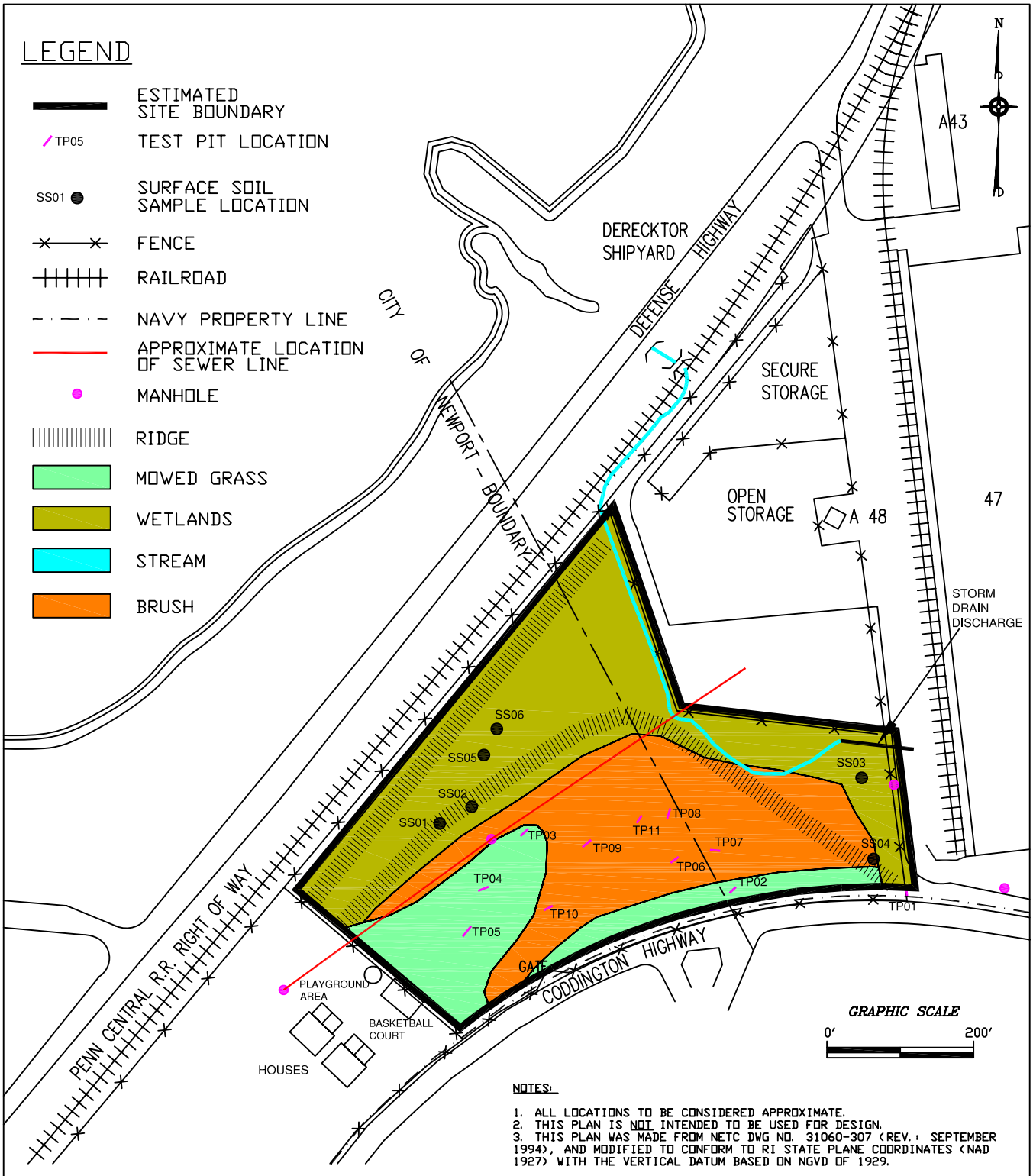


QUADRANGLE LOCATION

SITE LOCUS	FIGURE 10-1
CODDINGTON COVE RUBBLE FILL AREA	
NAVSTA NEWPORT – NEWPORT, RHODE ISLAND	

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	T. CAMPBELL	DATE:	MARCH 04, 2010
SCALE:	AS NOTED	ACAD NAME:	\02300\PP.DR\CCRF_LOCUS.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658-7899



NOTES:

1. ALL LOCATIONS TO BE CONSIDERED APPROXIMATE.
2. THIS PLAN IS NOT INTENDED TO BE USED FOR DESIGN.
3. THIS PLAN WAS MADE FROM NETC DWG NO. 31060-307 (REV.: SEPTEMBER 1994), AND MODIFIED TO CONFORM TO RI STATE PLANE COORDINATES (NAD 1927) WITH THE VERTICAL DATUM BASED ON NGVD OF 1929.

2005 PA INVESTIGATION SAMPLE LOCATIONS

CODDINGTON COVE RUBBLE FILL AREA

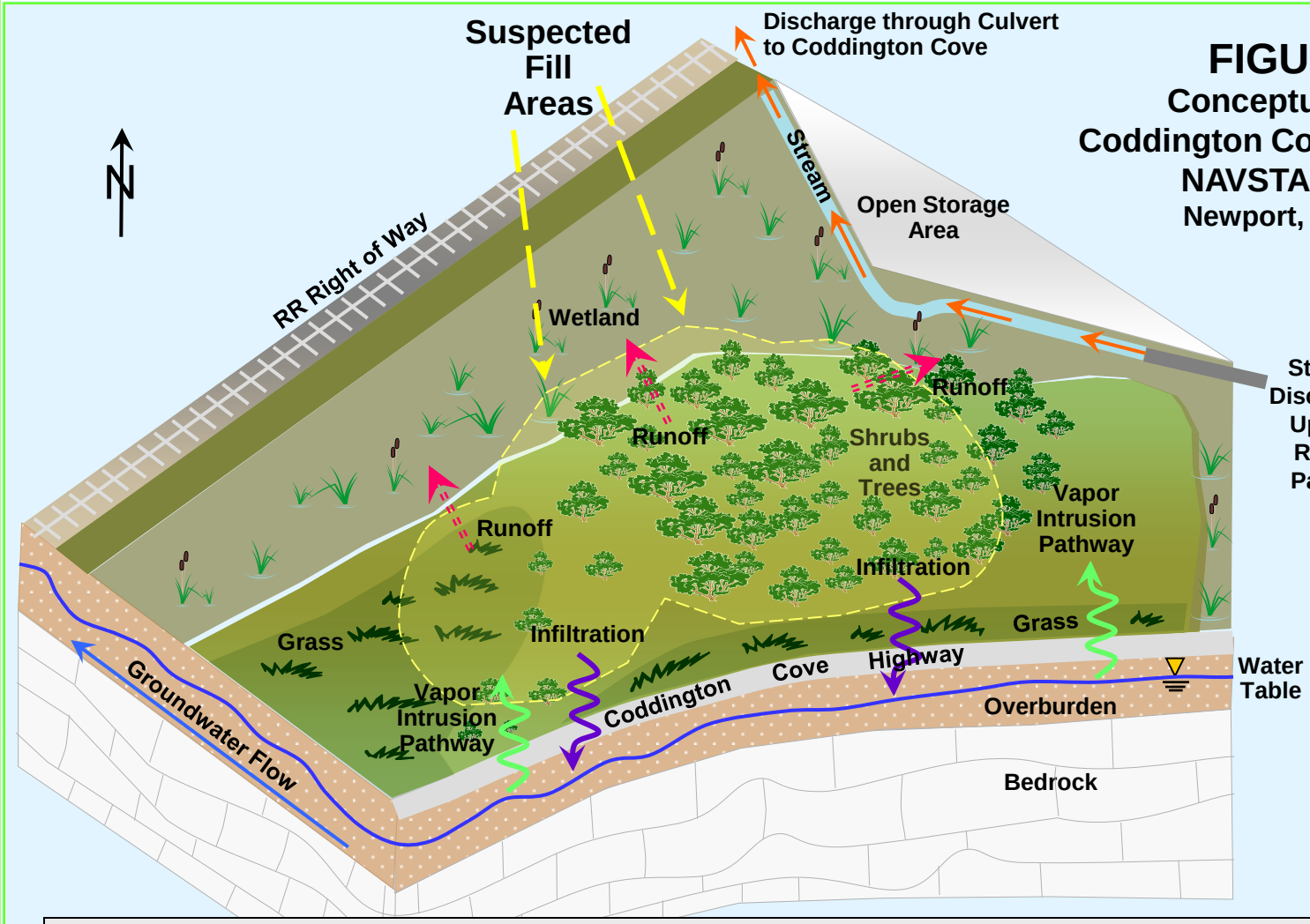
NAVSTA NEWPORT – NEWPORT, RHODE ISLAND

FIGURE 10-2

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	T. CAMPBELL	DATE:	MARCH 04, 2010
SCALE:	AS NOTED	ACAD NAME:	\02300\PP.DR\CCRF_PREV_SAMP.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658-7899

FIGURE 10-3
Conceptual Site Model
Coddington Cove Rubble Fill Area
NAVSTA NEWPORT
Newport, Rhode Island



Potential Exposure Pathways and Receptors

GW Soil
Onsite Resident
- Inhalation/ingestion of and/or dermal contact with surface and subsurface soils and sediments and surface water
- Groundwater ingestion

GW Sed SW
Off-Site Resident
- Groundwater ingestion
- Ingestion of and/or dermal contact with sediments and surface water

Soil Sed
Onsite Construction Worker
- Inhalation/ingestion and/or dermal contact with surface and subsurface soils and sediments and surface water

GW Soil Sed SW
Onsite Commercial Worker
- Inhalation/ingestion of and/or dermal contact with surface soil
- Groundwater ingestion and inhalation

Soil Sed SW
Wildlife (Aquatic/Terrestrial)
- Ingestion of vegetation, soil, and surface water and sediment
- Dermal contact with surface water, and sediment

Soil Sed SW
Onsite Trespasser
- Inhalation/ingestion of and/or dermal contact with surface soil
- Ingestion of and/or dermal contact with sediments and surface water

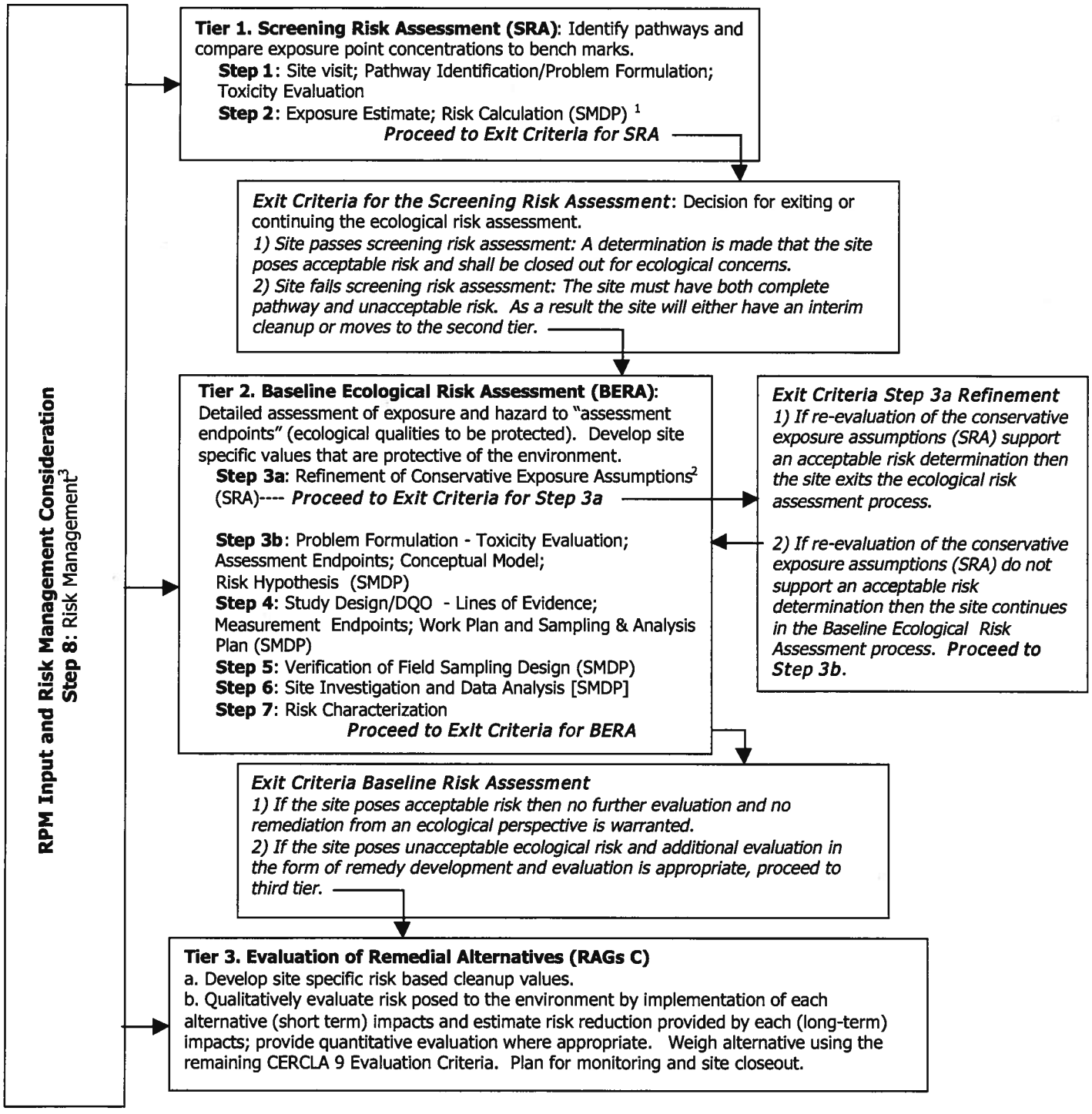
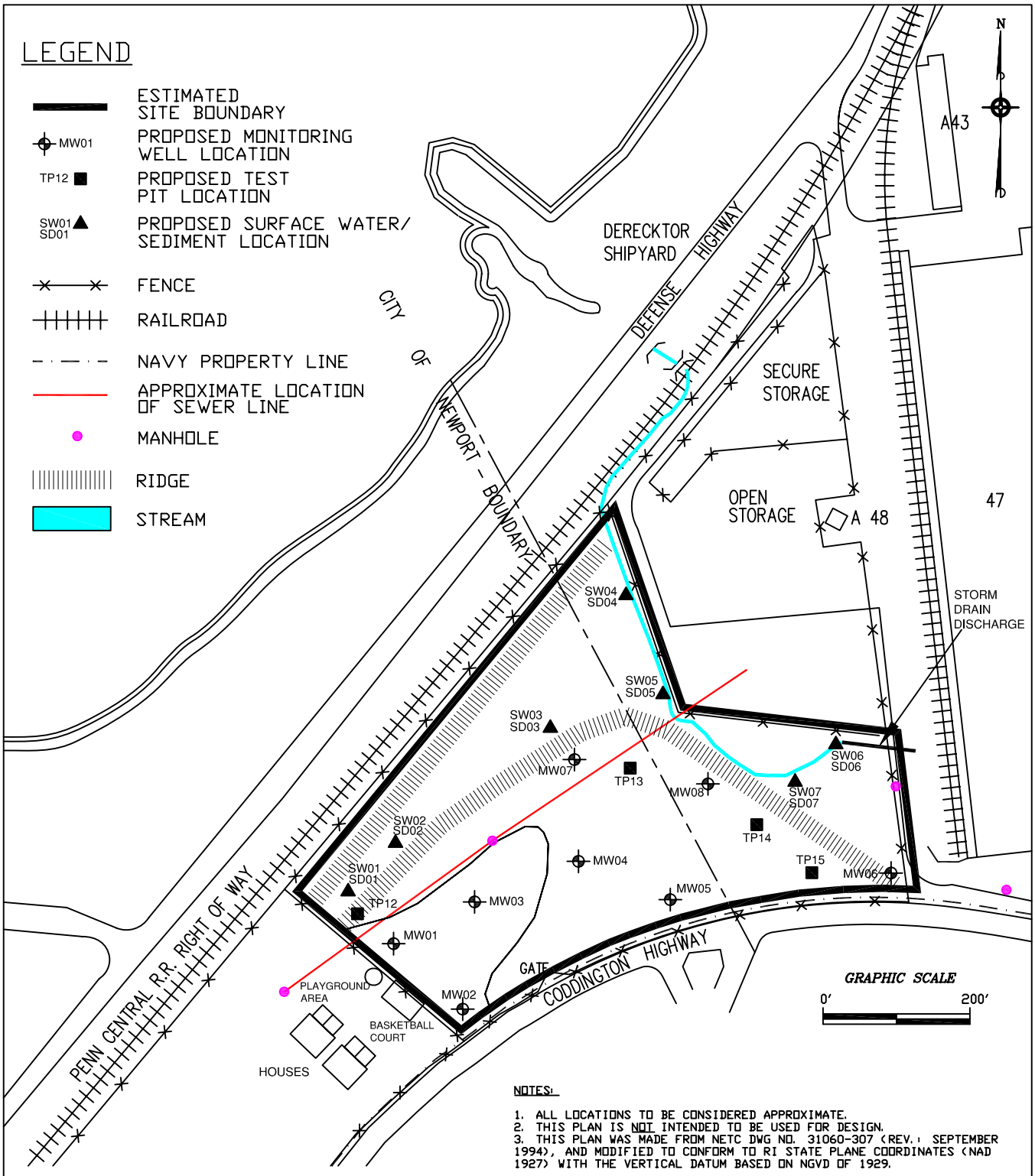


Figure 1. Navy Ecological Risk Assessment Tiered Approach

Notes: 1) See EPA's 8 Step ERA Process for requirements for each Scientific Management Decision Point (SMDP).
2) Refinement includes but is not limited to background, bioavailability, detection frequency, etc.
3) Risk Management is incorporated throughout the tiered approach.



NOTES:

1. ALL LOCATIONS TO BE CONSIDERED APPROXIMATE.
2. THIS PLAN IS NOT INTENDED TO BE USED FOR DESIGN.
3. THIS PLAN WAS MADE FROM NETC DWG NO. 31060-307 (REV.: SEPTEMBER 1994), AND MODIFIED TO CONFORM TO RI STATE PLANE COORDINATES (NAD 1927) WITH THE VERTICAL DATUM BASED ON NGVD OF 1929.

PROPOSED SAMPLE LOCATIONS

CODDINGTON COVE RUBBLE FILL AREA

NAVSTA NEWPORT – NEWPORT, RHODE ISLAND

FIGURE 17–1

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	T. CAMPBELL	DATE:	MARCH 04, 2010
SCALE:	AS NOTED	ACAD NAME:	\02300\PP.DR\CCRF_PROP_SAMP.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658-7899

REFERENCES

REFERENCES

Buchman, M. F., 2008. NOAA Screening Quick Reference Tables, NOAA OR&R Report 08-1, Seattle, WA, Office of Response and Restoration Division, National Oceanic and Atmospheric Administration. http://response.restoration.noaa.gov/cpr/sediment/squirt/squirt.html

CCME, 1997. Recommended Canadian Soil Quality Guidelines. Canadian Council of Ministers of the Environment. Ottawa, Ontario. March.

DON (Department of Navy), 1999. Navy Policy for Conducting Ecological Risk Assessments.

Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997a. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Oak Ridge National Laboratory. November. ES/ER/TM-85/R3

Efroymson, R.A., M.E. Will, and G.W. Suter II. 1997b. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Oak Ridge National Laboratory. November. ES/ER/TM-126/R2.

Envirodyne Engineers, Inc. (EEI). 1983. Initial Assessment Study of the Naval Education and Training Center, Newport, Rhode Island. Contract No. N62474-82-C-8262. Naval Energy and Environmental Support Activity (NEESA) 13-024. March.

Jones, D.S., R.N. Hull, and G.W. Suter II, 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota: 1997 Revision. Risk Assessment Program, Health Sciences Division, Oak Ridge, Tennessee. ES/ER/TM-95/R4. November.

Long, E.R., and L.G. Morgan, 1991. Potential for Biological Effects of Sediment-Sorbed Contaminants Tested in the National Status and Trends Program, National Ocean Service, Office of Oceanography and Marine Assessment, Rockville, Maryland, NOAA/TM/NOS/OMA-52.

MacDonald, D.D., C.G. Ingersoll, and T.A. Berger, 2000. "Development and Evaluation of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems." Archives of Environmental Contamination and Toxicology, Vol. 39, pp. 20-31.

Persaud, D., R. Jaagumagi, and A. Hayton, 1993. Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario. Ontario Ministry of Environment and Energy. August.

RIDEM (Rhode Island Department of Environmental Management), 2004. Rules and Regulations for the Investigation of Hazardous Material Releases, February.
http://www.dem.ri.gov/pubs/regs/regs/waste/remreg04.pdf

RIDEM (Rhode Island Department of Environmental Management), 2009. RIDEM Ambient Water Quality Criteria and Guidelines. July 2006, amended May 2009.

Suter, G.W. II. and C.L. Tsao, 1996. Toxicological Benchmarks for Screening Potential Constituents of Concern for Effects on Aquatic Biota:1996 Revision. Environmental Sciences Division, Oak Ridge National Laboratory. ES/ER/TM-96/R2.

Tetra Tech NUS, 2004. Technical Memorandum Record Review and Field Sampling Plan for Coddington Cove Rubble Fill Area (Study Area 04), Naval Station Newport, Newport, Rhode Island. May 11.

Tetra Tech NUS, 2005. Results of Field Sampling Effort – May and Junly 2004, Codington Cove Rubble Fill Area, SA-04, Naval Station Newport, Rhode Island. April 14.

USEPA (U.S. Environmental Protection Agency), 1996. ECO Update, Ecotox Thresholds. U.S. Environmental Protection Agency. Office of Solid Waste and Emergency Response. Intermittent Bulletin, Volume 3, Number 2. EPA540/F-95/038. January.

USEPA, 1997. Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments.

USEPA, 1998. Final Guidelines for Ecological Risk Assessment [United States Environmental Protection Agency (USEPA).

USEPA (U.S. Environmental Protection Agency), 2001. Region 4 Ecological Risk Assessment Bulletins – Supplement to RAGS. Effective April 20.
http://www.epa.gov/region04/waste/ots/ecolbul.htm

USEPA (U.S. Environmental Protection Agency), 2003. Ecological Soil Screening Level for Aluminum, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-60. November.

USEPA (U.S. Environmental Protection Agency), 2003a. Ecological Soil Screening Level for Iron, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-69. November.

USEPA, 2003b. Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: PAH Mixtures. EPA/600/R-02/013. November.

USEPA (U.S. Environmental Protection Agency), 2003c. Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: PAH Mixtures. EPA-600-R-02-013. Office of Research and Development. Washington, DC 20460.

USEPA (U.S. Environmental Protection Agency), 2005. Ecological Soil Screening Level for Antimony, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-61. February.

USEPA (U.S. Environmental Protection Agency), 2005. Ecological Soil Screening Level for Arsenic, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-62. March.

USEPA (U.S. Environmental Protection Agency), 2005. Ecological Soil Screening Level for Barium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-63. February.

USEPA (U.S. Environmental Protection Agency), 2005. Ecological Soil Screening Level for Beryllium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-64. February.

USEPA (U.S. Environmental Protection Agency), 2005. Ecological Soil Screening Level for Cadmium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-65. March.

USEPA (U.S. Environmental Protection Agency), 2005. Ecological Soil Screening Level for Chromium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-66. March.

USEPA (U.S. Environmental Protection Agency), 2005. Ecological Soil Screening Level for Cobalt, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-67. March.

USEPA (U.S. Environmental Protection Agency), 2005. Ecological Soil Screening Level for Lead, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-70. March.

USEPA (U.S. Environmental Protection Agency), 2005. Ecological Soil Screening Level for Vanadium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-75. April.

USEPA (U.S. Environmental Protection Agency), 2006. Ecological Soil Screening Level for Silver, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-77. October.

USEPA, 2006. EPA Region III BTAG, Freshwater Screening Benchmarks.
U.S. EPA (U.S. Environmental Protection Agency), 2008. EPA Region 9 Preliminary Remediation Goals (Revised October 2004). http://www.epa.gov/region09/waste/sfund/prg/index.html

USEPA (U.S. Environmental Protection Agency), 2007. Ecological Soil Screening Level for Copper, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-68. February.

USEPA (U.S. Environmental Protection Agency), 2007. Ecological Soil Screening Level for Nickel, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-76. March.

USEPA (U.S. Environmental Protection Agency), 2007. Ecological Soil Screening Level for DDT, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-57. April.

USEPA (U.S. Environmental Protection Agency), 2007. Ecological Soil Screening Level for Manganese, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-71. April.

USEPA (U.S. Environmental Protection Agency), 2007. Ecological Soil Screening Level for PAHs, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-78. June.

USEPA (U.S. Environmental Protection Agency), 2007. Ecological Soil Screening Level for Pentachlorophenol, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-58. April.

USEPA (U.S. Environmental Protection Agency), 2007. Ecological Soil Screening Level for Dieldrin, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-57. October.

U.S. EPA (U.S. Environmental Protection Agency), 2007. Ecological Soil Screening Level for Selenium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-72. November.

U.S. EPA (U.S. Environmental Protection Agency), 2007. Ecological Soil Screening Level for Zinc, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-73. November.

USEPA (U.S. Environmental Protection Agency), 2009. National Recommended Water Quality Criteria, Office of Water.

USEPA, 2009. Regional Screening Level (RSL) Master Table. January 7. http://www.epa.gov/region09/superfund/prg/rsl-table.html

APPENDIX A

TETRA TECH AND USEPA SAMPLING AND RELATED STANDARD OPERATING PROCEDURES

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number GH-1.5	Page 1 of 20
		Effective Date 06/99	Revision 1
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject BOREHOLE AND SAMPLE LOGGING		Approved D. Senovich	

TABLE OF CONTENTS

SECTION		PAGE
1.0	**PURPOSE**	**3**
2.0	**SCOPE**	**3**
3.0	**GLOSSARY**	**3**
4.0	**RESPONSIBILITIES**	**3**
5.0	**PROCEDURES**	**3**
5.1	MATERIALS NEEDED	3
5.2	CLASSIFICATION OF SOILS	3
5.2.1	USCS Classification	6
5.2.2	Color	6
5.2.3	Relative Density and Consistency	6
5.2.4	Weight Percentages	7
5.2.5	Moisture	10
5.2.6	Stratification	10
5.2.7	Texture/Fabric/Bedding	10
5.2.8	Summary of Soil Classification	10
5.3	CLASSIFICATION OF ROCKS	13
5.3.1	Rock Type	13
5.3.2	Color	16
5.3.3	Bedding Thickness	16
5.3.4	Hardness	16
5.3.5	Fracturing	16
5.3.6	Weathering	17
5.3.7	Other Characteristics	17
5.3.8	Additional Terms Used in the Description of Rock	18
5.4	ABBREVIATIONS	19
5.5	BORING LOGS AND DOCUMENTATION	19
5.5.1	Soil Classification	19
5.5.2	Rock Classification	23
5.5.3	Classification of Soil and Rock from Drill Cuttings	24
5.6	REVIEW	24
6.0	**REFERENCES**	**24**
7.0	**RECORDS**	**25**

TABLE OF CONTENTS (Continued)

FIGURES

NUMBERS **PAGE**

1 BORING LOG (EXAMPLE) 4
2 CONSISTENCY FOR COHESIVE SOILS 8
3 BEDDING THICKNESS CLASSIFICATION 10
4 GRAIN SIZE CLASSIFICATION FOR ROCKS 12
5 COMPLETED BORING LOG (EXAMPLE) 17

1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

FIGURE 1
BORING LOG (EXAMPLE)

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

Remarks: _____

Drilling Area Background (ppm): ☐

Converted to Well: Yes _____ No _____ Well I.D. #: _____

FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)

COURSE-GRAINED SOILS More Than Half of Material is LARGER Than No. 200 Sieve Size					FINE-GRAINED SOILS More Than Half of Material is SMALLER Than No. 200 Sieve Size					
FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 inches and Basing Fractions on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES	FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 inches and Basing Fractions on Estimated Weights)				GROUP SYMBOL	TYPICAL NAMES
					Identification Procedures on Fraction Smaller than No. 40 Sieve Size					
						DRY STRENGTH (Crushing Characteristics)	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)		
GRAVELS (More than half of coarse fraction is larger than No. 4 sieve size)	CLEAN GRAVELS (Less than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well graded gravels, gravel-sand mixtures, little or no fines.	SILTS AND CLAYS Liquid Limit less than 50	None to Slight	Quick to Slow	None	ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines.		Medium to High	None to Very Slow	Medium	CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	GRAVELS WITH FINES (More than 12% Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.		Slight to Medium	Slow	Slight	OL	Organic silts and organic silty clays of low plasticity.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.	SILTS AND CLAYS Liquid Limit greater than 50	Slight to Medium	Slow to None	Slight to Medium	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
SANDS (50% or more of coarse fraction is smaller than No. 4 sieve size)	CLEAN SANDS (Less than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sand, gravelly sands, little or no fines.		High to Very High	None	High	CH	Inorganic clays of high plasticity, fat clays.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.		Medium to High	None to Very Slow	Slight to Medium	OH	Organic clays of medium to high plasticity.
	SANDS WITH FINES (More than 12% Fines)	Non-plastic fines (for identification procedures, see MCL)	SM	Silty sands, poorly graded sand-silt mixtures.	HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture.			PT	Peat and other organic soils
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.						

Boundary classifications: Soils possessing characteristics of two groups are designated by combining group symbols. For example, GW-GC, well graded gravel-sand mixture with clay binder.
All sieve sizes on this chart are U.S. Standard.

DENSITY OF GRANULAR SOILS	
DESIGNATION	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT
Very Loose	0-4
Loose	5-10
Medium Loose	11-30
Dense	31-50
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS			
CONSISTENCY	UNCONFINED COMPRESSIVE STRENGTH (TONS/SQ. FT.)	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT	FIELD IDENTIFICATION METHODS
Very Soft	Less than 0.25	0 to 2	Easily penetrated several inches by fist
Soft	0.25 to 0.50	2 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.50 to 1.0	4 to 8	Can be penetrated several inches by thumb.
Stiff	1.0 to 2.0	8 to 15	Readily indented by thumb.
Very Stiff	2.0 to 4.0	15 to 30	Readily indented by thumbnail.
Hard	More than 4.0	Over 30	Indented with difficulty by thumbnail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)			ROCK BROKENNESS		
Descriptive Terms	Scratcher or Knife Blade	Hammer Blade	Descriptive Terms	Abbreviation	Spacing
Soft	Easily Gouged	Crushes when pressed with hammer	Very Broken	(V.Br.)	0"
Medium Soft	Can be Gouged	Breaks (one blow); crumbly edges	Broken	(Br.)	2"-1'
Medium Hard	Can be scratched	Breaks (one blow); sharp edges	Blocky	(Bl.)	1'+
Hard	Cannot be scratched	Breaks conchoidally (several blows); sharp edges	Massive	(M)	3'+

LEGEND :

SOIL SAMPLES - TYPES

S-2" Split Barrel Sample

ST-3" O.D. Undisturbed Sample

O - Other Samples, Specify in Remarks

ROCK SAMPLES - TYPES

X-NX (Conventional) Core (2.1-1/4" O.D.)

Q-NQ (Q-Wireline) Core (1.89-1.41" O.D.)

Z - Other Core Sizes, Specify in Remarks

WATER LEVELS

Ø/P

Initial Level w/Date & Depth

Ø/P

Stabilized Level w/Date & Depth

5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).
This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

FIGURE 2

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

5.2.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

FIGURE 3

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ($CaCO_3$). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

FIGURE 4

GRAIN SIZE CLASSIFICATION FOR ROCKS

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).

- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.

- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.

- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the works "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

$$RQD\ \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C	-	Coarse	Lt	-	Light	Yl	-	Yellow
Med	-	Medium	BR	-	Broken	Or	-	Orange
F	-	Fine	BL	-	Blocky	SS	-	Sandstone
V	-	Very	M	-	Massive	Sh	-	Shale
Sl	-	Slight	Br	-	Brown	LS	-	Limestone
Occ	-	Occasional	Bl	-	Black	Fgr	-	Fine-grained
Tr	-	Trace						

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.

- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.

- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.

- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.

- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5
COMPLETED BORING LOG (EXAMPLE)

BOREHOLE LOG

Page 1 of 1

PROJECT NAME: NSB - SITE
PROJECT NUMBER: 9594
DRILLING COMPANY: SOILTEST CO.
DRILLING RIG: CME-55
BORING NUMBER: SB/MW 1
DATE: 3/8/96
GEOLOGIST: SJ CONTI
DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**
S-1	0.0	7/6	1.5/2.0		M DENSE	BRN	SILTY SAND - SOME	SM	MOIST SL. ORG. ODOR	5	0	0	0
C 0800	2.0	9/10				TO BLK	ROCK FR - TR BRICKS (FILL)		FILL TO 4'±				
	4.0			4.0									
S-2 C		5/7	2.9/2.0		M DENSE	BRN	SILTY SAND - TR FINE GRAVEL	SM	MOIST - W ODOR	10	10	-	-
0810	6.0	9/8							NAT. MATL. TOOK SAMPLE SB01-0406 FOR ANALYSIS				
	•			7' ▽									
	8.0			8.0									
S-3 C		6/8	1.9/2.0		DENSE	TAN BRN	FINE TO COARSE SAND	SW	WET	0	0	0	0
0820	10.0	17/16					TR. F. GRAVEL		HIT WATER @ 7'±				
	12.0			12.0									
S-4 C		7/6	1.6/2.0		STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	-5	-	-
0830	14.0	5/8											
	15.0			15.0					AUGER REF @ 15'				
				LITHIE 16	M HARD	BRN	SILTSTONE	VBR	WEATHERED				
9/5	①		4.0/5.0						LO # JNTS @ 15.5 WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
				19'					LOSING SOME				
	20.0				HARD	GRAY	SANDSTONE - SOME SILTSTONE	BR	DRILL H20 @ 17'±				
									SET TEMP 6" CAS TO 15.5				
4.9/5.0	②		5.0/5.0						SET 2" Ø PVC SCREEN 16-25	0	0	0	0
									SAND 14-25				
	25.0			25					PELLETS 12-14				

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

• 1-20Z
1-80Z

Drilling Area Background (ppm): 0

Remarks: CME-55 R16, 4¼" ID HSA - 9" OD±
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP
NX CORE IN BEDROCK RUN ① = 25 min. RUN ② = 15 min

Converted to Well: Yes ✓ No ____ Well I.D. #: MW-1

- Enter color of the material in the appropriate column.
- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:
  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent
- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.
- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.
- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:
  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.
  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.
  - Particle shape - flat, elongated, or flat and elongated.
  - Maximum particle size or dimension.
  - Water level observations.
  - Reaction with HCl - none, weak, or strong.
- Additional comments:
  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.
  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.
  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.
  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.

- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.

- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.

- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.

- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.

- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:

  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.

- Remarks at the bottom of Boring Log shall include:

  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.

- As a final check the boring log shall include the following:

  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.

  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number CT-04	Page 1 of 7
		Effective Date 03/09/09	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SAMPLE NOMENCLATURE		Approved Tom Johnston	

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE 2

2.0 SCOPE 2

3.0 GLOSSARY 2

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS 2

5.0 PROCEDURES 3

5.1 INTRODUCTION 3
5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS 3
5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS 4
5.4 EXAMPLES OF SAMPLE NOMENCLATURE 5
5.5 FIELD QA/QC SAMPLE NOMENCLATURE 6
5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE 6

6.0 DEVIATIONS 7

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix
- Sorting of data by depth
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints (maximum of 20 characters)

2.0 SCOPE

The methods described in this SOP shall be used consistently for all projects requiring electronic data. Other contract- or project-specific sample nomenclature requirements may also be applicable.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Program Manager - It shall be the responsibility of the Project Manager (or designee) to inform contract-specific Project Managers (PMs) of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of the PM to determine the applicability of this SOP based on: (1) program-specific requirements and (2) project size and objectives. It shall be the responsibility of the PM (or designee) to ensure that sample nomenclature requirements are thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and are consistent with this SOP if relevant. It shall be the responsibility of the PM to ensure that the FOL is familiar with the sample nomenclature system.

Field Operations Leader (FOL) - It shall be the responsibility of the FOL to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP and the project-specific sample nomenclature system. It shall be the responsibility of the FOL to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

General personnel qualifications for sample nomenclature activities in the field include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for field documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 INTRODUCTION

The sample identification (ID) system can consist of as few as eight but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the laboratory has three segments and shall be as follows, where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3 or 4 Characters	**AAA 2 or 3 Characters**	**A or N 3 to 6 Characters**
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

(1) Soil and sediment sample ID

A or N 3 or 4 Characters	**AAA 2 or 3 Characters**	**A or N 3 to 6 Characters**	**NNNN 4 Characters**
Site identifier	Sample type	Sample location	Sample depth

(2) Aqueous (groundwater or surface water) sample ID

A or N 3 or 4 Characters	**AAA 2 or 3 Characters**	**A or N 3 to 6 Characters**	**NN 2 Characters**	**-A 1 Character**
Site identifier	Sample type	Sample location	Round number	Filtered sample only

(3) Biota sample ID

A or N 3 or 4 Characters	**AAA 2 or 3 Characters**	**A or N 3 to 6 Characters**	**AA 2 Characters**	**NNN 3 Characters**
Site identifier	Sample type	Sample location	Species identifier	Sample group number

5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS

The various fields in the sample ID include but are not limited to the following:

- Site identifier
- Sample type
- Sample location
- Sample depth
- Sampling round number
- Filtered
- Species identifier
- Sample group number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary because many facilities/sites have multiple individual sites, Solid Waste Management Units (SWMUs), Operable Units (OUs), etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six characters (alpha, numeric, or a mixture). The six characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to three characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet or boring log, in the logbook, etc.

A two-digit round number will be used to track the number of aqueous samples collected from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three-digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001, and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS

Examples of each of the fields are as follows:

Site identifier - Examples of site numbers/designations are as follows:

A01 - Area of Concern (AOC) 1
125 - SWMU 125
000 - Base- or facility-wide sample (e.g., upgradient well)
BBG - Base background

The examples cited are only suggestions. Each PM (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample type - Examples of sample types are as follows:

AH - Ash Sample

AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation-Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample
SG - Soil Gas Sample
SL - Sludge Sample
SP - Seep Sample
SS - Surface Soil Sample
ST - Storm Sewer Water Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WP - Wipe Sample
WS - Waste/Solid Sample
WW - Wastewater Sample

Sample location - Examples of the location field are as follows:

001 - Monitoring well 1
N32E92 - Grid location 32 North and 92 East
D096 - Investigation-derived waste drum number 96

Species identifier - Examples of species identifier are as follows:

BC - Blue Crab
GB - Blue Gill
CO - Corn
SB - Soybean

5.4 EXAMPLES OF SAMPLE NOMENCLATURE

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full-body analysis, the first time a minnow trap was checked at grid location A25 of SWMU 1415, three small blue gills were captured, collected, and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415), the sample ID would be 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash (-F).

5.5 FIELD QA/QC SAMPLE NOMENCLATURE

Field Quality Assurance (QA)/Quality Control (QC) samples are designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC type	Date	Sequence number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
WB = Source Water Blank

The sampling time recorded on the chain-of-custody form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log Sheet (see SOP SA-6.3, Field Documentation).

5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE

The first duplicate of the day for a filtered groundwater sample collected on June 3, 2000, would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003, would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000, would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001, would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site-specific planning documents.

STANDARD OPERATING PROCEDURE
CALIBRATION OF FIELD INSTRUMENTS
(temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity)

Quality Assurance Unit
U.S. Environmental Protection Agency – Region 1
11 Technology Drive
North Chelmsford, MA 01863

The controlled version of this document is the electronic version viewed on-line only. If this is a printed copy of the document, it is an uncontrolled version and may or may not be the version currently in use.

This document contains direction developed solely to provide guidance to U.S. Environmental Protection Agency (EPA) personnel. EPA retains the discretion to adopt approaches that differ from these procedures on a case-by-case basis. The procedures set forth do not create any rights, substantive or procedural, enforceable at law by party to litigation with EPA or the United States.

Prepared by: Charles Porfert 1/19/10
(Charles Porfert, Quality Assurance Unit) Date

Approved by: Gerard Sotolongo 1-19-10
(Gerard Sotolongo, Quality Assurance Unit) Date

Revision Page

Date	Rev #	Summary of changes	Sections
6/03/98	1	Draft	
01/19/10	2	Finalized	

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION....4

2.0 HEALTH AND SAFETY WARNINGS....4

3.0 GENERAL....4

4.0 FREQUENCY OF CALIBRATION....5

5.0 CALIBRATION PROCEDURES....5

5.1 TEMPERATURE....6

5.2 pH (electrometric)....7

5.3 DISSOLVED OXYGEN....8

5.4 SPECIFIC CONDUCTANCE....10

5.5 OXIDATION/REDUCTION POTENTIAL (ORP)....11

5.6 TURBIDITY....12

6.0 POST CALIBRATION CHECK....14

7.0 DATA MANAGEMENT AND RECORDS MANAGEMENT....15

8.0 REFERENCES....15

Table 1 INSTRUMENT CALIBRATION LOG....16

OXYGEN SOLUBILITY....17

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to provide a framework for calibrating field instruments used to measure water quality parameters for groundwater and surface water. Water quality parameters include temperature, pH, dissolved oxygen, specific conductance, oxidation/reduction potential [ORP], and turbidity. This SOP supplements, but does not replace, EPA analytical methods listed in 40 CFR 136 and 40 CFR 141 for temperature, dissolved oxygen, conductivity/specific conductance, pH and turbidity.

This SOP is written for instruments that measure temperature, pH, dissolved oxygen, specific conductance, turbidity, and/or oxidation/reduction potential [ORP] and the probe readings for pH, dissolved oxygen, and specific conductance are automatically corrected for temperature.

For groundwater monitoring, the instrument must be equipped with a flow-through-cell and the display/logger or computer display screen needs to be large enough to simultaneously contain the readouts of each probe in the instrument. Turbidity is measured using a separate instrument. It must not be measured in a flow-through-cell because the flow-through-cell acts as a sediment trap. This procedure is applicable for use with the *EPA Region 1 Low Stress (low flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells.*

2.0 HEALTH AND SAFETY WARNINGS

Read all labels on the standards and note any warnings on the labels. Wear appropriate personal protection equipment (e.g., gloves, eye shields, etc.) when handling the standards. If necessary, consult the Material Safety Data Sheets (MSDS) for additional safety information on the chemicals in the standards.

3.0 GENERAL

All monitoring instruments must be calibrated before they are used to measure environmental samples. For instrument probes that rely on the temperature sensor (pH, dissolved oxygen, specific conductance, and oxidation/reduction potential [ORP]), each temperature sensor needs to be checked for accuracy against a thermometer that is traceable to the National Institute of Standards and Technology (NIST). Before any instrument is calibrated or used to perform environmental measurements, the instrument must stabilize (warm-up) according to manufacturer's instructions and must have no air bubbles lodged between the probe and probe guard.

Most projects will require at least two standards to bracket the expected measurement range. This means that one standard is less than the expected value and one is higher. When an environmental sample measurement falls outside the calibration range, the instrument must be re-calibrated to bracket the new range before continuing measurements. Otherwise, the measurements that are outside the calibration range will need to be qualified.

This SOP requires that the manufacturer's instruction manual (including the instrument specifications) accompany the instrument into the field.

4.0 FREQUENCY OF CALIBRATION

At a minimum, the instrument is calibrated prior to use on the day the measurements are to be performed. A post calibration check at the end of the day is performed to determine if the instrument drifted out of calibration. Some projects may require more frequent calibration checks throughout the day in addition to the check at the end of the day. For these checks, the instrument can be recalibrated during the day if the instrument drifted out of calibration and only the data measured prior to the check would need to be qualified. The calibration/post calibration data information is recorded in Table 1.

Instruments (e.g., sonde) that monitor continuously over a period of time are calibrated before deployment. When these instruments are recovered, the calibration is checked to determine if any of them drifted out of calibration.

Some instruments lose their calibration criteria when they are turned off. Those instruments can either be left on all day (battery dependent) or calibrated at each sampling location. If they are calibrated at each sampling location, a post calibration check is not needed.

Ideally, the temperature of the standards should be close to the temperature of the ambient water that is being measured.

5.0 CALIBRATION PROCEDURES

Prior to calibration, all instrument probes and cable connections must be cleaned and the battery checked according to the manufacturer's instructions. Failure to perform these steps (proper maintenance) can lead to erratic measurements.

If a multi-probe instrument is to be used, program the instrument to display the parameters to be measured (e.g., temperature, pH, percent dissolved oxygen, mg/L dissolved oxygen, specific conductance, and ORP).

The volume of the calibration solutions must be sufficient to cover both the probe and temperature sensor (see manufacturer's instructions for the volume to be used).

Check the expiration date of the standards. Do not use expired standards.

All standards are stored according to manufacturer instructions.

5.1 TEMPERATURE

Most instrument manuals state there is no calibration of the temperature sensor, but the temperature sensor must be checked to determine its accuracy. This accuracy check is performed at least once per year and the accuracy check date/information is kept with the instrument. If the accuracy check date/information is not included with the instrument or the last check was over a year, the temperature sensor accuracy needs to be checked at the beginning of the sampling event. If the instrument contains multiple temperature sensors, each sensor must be checked. This procedure is not normally perform in the field. If the instrument is obtained from a rental company, the rental company should performed the calibration check and include with the instrument documentation that it was performed.

Verification Procedure

1. Fill a container with water and adjust the water temperature to below the water body's temperature to be measured. Use ice or warm water to adjust the temperature.

2. Place a thermometer that is traceable to the National Institute of Standards and Technology (NIST) and the instrument's temperature sensor into the water. Wait for both temperature readings to stabilize.

3. Compare the two measurements. The instrument's temperature sensor must agree with the reference thermometer measurement within the accuracy of the sensor (e.g., $\pm 0.2\,^{\circ}C$). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

4. Adjust the water temperature to a temperature higher than the water body to be measured.

5. Compare the two measurements. The instrument's temperature sensor must agree with the reference thermometer measurement within the accuracy of the sensor (e.g.,

±0.2°C). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

5.2 pH (electrometric)

The pH of a sample is determined electrometrically using a glass electrode.

Choose the appropriate buffered standards that will bracket the expected values at the sampling locations. If the water body's pH is unknown, then three standards are needed for the calibration: one close to seven, one at least two pH units below seven, and the other at least two pH units above seven. Instruments that will not accept three standards will need to be re-calibrated if the water sample's pH is outside the initial calibration range described by the two standards.

Calibration Procedure

1. Allow the buffered standards to equilibrate to the ambient temperature.

2. Fill calibration containers with the buffered standards so each standard will cover the pH probe and temperature sensor.

3. Remove probe from its storage container, rinse with deionized water, and remove excess water.

4. Select measurement mode. Immerse probe into the initial standard (e.g., pH 7).

5. Wait until the readings stabilize. If the reading does not change within 30 seconds, select calibration mode and then select "pH". Enter the buffered standard value into instrument.

6. Remove probe from the initial standard, rinse with deionized water, and remove excess water.

7. Immerse probe into the second standard (e.g., pH 4). Repeat step 5.

8. Remove probe from the second standard, rinse with deionized water, and remove excess water. If instrument only accepts two standards, the calibration is complete. Go to step 11. Otherwise continue.

9. Immerse probe in third buffered standard (e.g., pH 10) and repeat step 5.

10. Remove probe from the third standard, rinse with deionized water, and remove excess water.

11. Select measurement mode, if not already selected. To ensure that the initial calibration standard (e.g., pH 7) has not changed, immerse the probe into the initial standard. Wait for the readings to stabilize. The reading should read the initial standard value within the manufacturer's specifications. If not, re-calibrate the instrument. If re-calibration does not help, the calibration range may be too great. Reduce calibration range by using standards that are closer together.

12. The calibration is complete. Rinse the probe with deionized water and store the probe according to manufacturer's instructions.

13. Record the calibration information on Table 1.

5.3 DISSOLVED OXYGEN

Dissolved oxygen (DO) content in water is measured using a membrane electrode. To insure proper operation, the DO probe's membrane and electrolyte should be replaced prior to calibration for the sampling event. The new membrane may need to be conditioned before it is used; consult manufacturer's manual on how the conditioning is to be performed. Failure to perform this step may lead to erratic measurements. Before performing the calibration/measurements, inspect the membrane for air bubbles and nicks.

Note: some manufacturers require an altitude correction instead of a barometric correction. In that case, enter the altitude correction according to the manufacturer's directions in Step 5 and then proceed to Step 6.

Note: some instruments have a built-in barometer. Follow the manufacturer's instructions for entering the barometric value in step 5.

Calibration Procedure

1. Gently dry the temperature sensor and remove any water droplets from the DO probe's sensor membrane according to manufacturer's instructions. Note that the evaporation of moisture on the temperature sensor or DO probe may influence the readings during calibration.

2. Create a 100 percent water-saturated air environment by placing a wet sponge or a

wet paper towel on the bottom of the DO calibration container. Place the DO probe into the calibration container. The probe is loosely fitted into the calibration container to prevent the escape of moisture evaporating from the sponge or paper towel while maintaining ambient pressure (see manufacturer's instructions). Note that the probe and the temperature sensor must not come in contact with these wet items.

3. Allow the confined air to become saturated with water vapor (saturation occurs in approximately 10 to 15 minutes). During this time, turn on the instrument to allow the DO probe to warm-up. Select the measurement mode. Check the temperature readings. Readings must stabilize before continuing to the next step.

4. Select calibration mode; then select "DO %".

5. Enter the local barometric pressure (usually in mm of mercury) for the sampling location into the instrument. This measurement must be determined from an on-site barometer. Do not use barometric pressure obtained from the local weather services unless the pressure is corrected for the elevation of the sampling location. [Note: inches of mercury times 25.4 mm/inch equals mm of mercury or consult Oxygen Solubility at Indicated Pressure chart attached to the SOP for conversion at selected pressures].

6. The instrument should indicate that the calibration is in progress. After calibration, the instrument should display percent saturated DO.

7. Select measurement mode and set the display to read DO mg/L and temperature. Compare the DO mg/L reading to the Oxygen Solubility at Indicated Pressure chart attached to the SOP. The numbers should agree. If they do not agree within the accuracy of the instrument (usually ± 0.2 mg/L), repeat calibration. If this does not work, change the membrane and electrolyte solution.

8. Remove the probe from the container and place it into a 0.0 mg/L DO solution (see footnote). Check temperature readings. They must stabilize before continuing.

9. Wait until the "mg/L DO" readings have stabilized. The instrument should read less than 0.5 mg/L (assuming an accuracy of ± 0.2 mg/L). If the instrument reads above 0.5 mg/L or reads negative, it will be necessary to clean the probe, and change the membrane and electrolyte solution. If this does not work, try a new 0.0 mg/L DO solution. If these changes do not work, contact the manufacturer. Note: some projects and instruments may have different accuracy requirements. The 0.5 mg/L

value may need to be adjusted based on the accuracy requirements of the project or instrument.

10. After the calibration has been completed, rinse the probe with tap or deionized water and store the probe according to manufacturer's instructions. It is important that all of the 0.0 mg/L DO solution be rinsed off the probe so as not to effect the measurement of environmental samples.

11. Record calibration information on Table 1.

Note: You can either purchase the 0.0 mg/L DO solution from a vendor or prepare the solution yourself. To prepare a 0.0 mg/L DO solution, follow the procedure stated in Standard Methods (Method 4500-O G). The method basically states to add excess sodium sulfite (until no more dissolves) and a trace amount of cobalt chloride (read warning on the label before use) to water. This solution is prepared prior to the sampling event. Note: this solution can be made without cobalt chloride, but the probe will take longer to respond to the low DO concentration.

5.4 SPECIFIC CONDUCTANCE

Conductivity is used to measure the ability of an aqueous solution to carry an electrical current. Specific conductance is the conductivity value corrected to 25 °C.

Most instruments are calibrated against a single standard which is near the specific conductance of the environmental samples. The standard can be either below or above the specific conductance of the environmental samples. A second standard is used to check the linearity of the instrument in the range of measurements.

When performing specific conductance measurement on groundwater or surface water and the measurement is outside the initial calibration range defined by the two standards, the instrument will need to be re-calibrated using the appropriate standards.

Specific Conductance Calibration Procedure

1. Allow the calibration standards to equilibrate to the ambient temperature.

2. Fill calibration containers with the standards so each standard will cover the probe and temperature sensor. Remove probe from its storage container, rinse the probe with deionized water or a small amount of the standard (discard the rinsate), and place the

probe into the standard.

3. Select measurement mode. Wait until the probe temperature has stabilized.

4. Select calibration mode, then specific conductance. Enter the specific conductance standard value. Make sure that the units on the standard are the same as the units used by the instrument. If not, convert the units on the standard to the units used by the instrument.

5. Select measurement mode. The reading should remain within manufacturer's specifications. If it does not, re-calibrate. If readings continue to change after re-calibration, consult manufacturer or replace calibration solution.

6. Remove probe from the standard, rinse the probe with deionized water or a small amount of the second standard (discard the rinsate), and place the probe into the second standard. The second standard will serve to verify the linearity of the instrument. Read the specific conductance value from the instrument and compare the value to the specific conductance on the standard. The two values should agree within the specifications of the instrument. If they do not agree, re-calibrate. If readings do not compare, then the second standard may be outside the linear range of the instrument. Use a standard that is closer to the first standard and repeat the verification. If values still do not compare, try cleaning the probe or consult the manufacturer.

7. After the calibration has been completed, rinse the probe with deionized water and store the probe according to manufacturer's instructions.

8. Record the calibration information on Table 1.

Note: for projects where specific conductance is not a critical measurement it may be possible to calibrate with one standard in the range of the expected measurement.

5.5 OXIDATION/REDUCTION POTENTIAL (ORP)

The oxidation/reduction potential is the electrometric difference measured in a solution between an inert indicator electrode and a suitable reference electrode. The electrometric difference is measured in millivolts and is temperature dependent.

Calibration or Verification Procedure

1. Allow the calibration standard (a Zobell solution: read the warning on the label before use) to equilibrate to ambient temperature.

2. Remove the probe from its storage container and place it into the standard.

3. Select measurement mode.

4. Wait for the probe temperature to stabilize, and then read the temperature.

5. If the instrument is to be calibrated, do Steps 6 and 7. If the instrument calibration is to be verified, then go to Step 8.

6. Look up the millivolt (mv) value at this temperature from the millivolt versus temperature correction table usually found on the standard bottle or on the standard instruction sheet. You may need to interpolate millivolt value between temperatures. Select "calibration mode", then "ORP". Enter the temperature-corrected ORP value into the instrument.

7. Select measurement mode. The readings should remain unchanged within manufacturer's specifications. If they change, re-calibrate. If readings continue to change after re-calibration, try a new Zobell solution or consult manufacturer. Go to Step 9.

8. If the instrument instruction manual states that the instrument is factory calibrated, then verify the factory calibration against the Zobell solution. If they do not agree within the specifications of the instrument, try a new Zobell solution. If it does not agree, the instrument will need to be re-calibrated by the manufacturer.

9. After the calibration has been completed, rinse the probe with deionized water and store the probe according to manufacturer's instructions.

10. Record the calibration information on Table 1.

5.6 TURBIDITY

The turbidity method is based upon a comparison of intensity of light scattered by a sample under defined conditions with the intensity of light scattered by a standard reference suspension. A

turbidimeter is a nephelometer with a visible light source for illuminating the sample and one or more photo-electric detectors placed ninety degrees to the path of the light source. Note: the below calibration procedure is for a turbidimeter which the sample is placed into a cuvette.

Some instruments will only accept one standard. For those instruments, the second, third, etc., standards will serve as check points.

Calibration Procedures

1. Allow the calibration standards to equilibrate at the ambient temperature. The use of commercially available polymer primary standards (AMCO-AEPA-1) is preferred; however, the standards can be prepared using Formazin (read the warning on the label before use) according to the EPA analytical Method 180.1. Other standards may be used if they can be shown that they are equivalent to the previously mentioned standards.

2. If the standard cuvette is not sealed, rinse a cuvette with deionized water. Shake the cuvette to remove as much water as possible. Do not wipe dry the inside of the cuvette because lint from the wipe may remain in the cuvette. Add the standard to the cuvette.

3. Before performing the calibration procedure, make sure the cuvettes are not scratched and the outside surfaces are dry and free from fingerprints and dust. If the cuvette is scratched or dirty, discard or clean the cuvette respectively. Note: some manufacturers require the cuvette to be orientated in the instrument in a particular direction for accurate reading.

4. Select a low value standard such as a zero or 0.02 NTU and calibrate according to manufacturer's instructions. Note: a zero standard (approximately 0 NTU) can be prepared by passing distilled water through a 0.45 micron pore size membrane filter.

5. Select a high standard and calibrate according to manufacturer's instructions or verify the calibration if instrument will not accept a second standard. In verifying, the instrument should read the standard value to within the specifications of the instrument. If the instrument has range of scales, check each range that will be used during the sampling event with a standard that falls within that range.

6. Record the calibration information on Table 1.

6.0 POST CALIBRATION CHECK

After the initial calibration is performed, the instrument's calibration may drift during the measurement period. As a result, you need to determine the amount of drift that occurred after collecting the measurements. This is performed by placing the instrument in measurement mode (not calibration mode) and placing the probe in one or more of the standards used during the initial calibration; for turbidity place the standard in a cuvette and then into the turbidimeter. Wait for the instrument to stabilize and record the measurement (Table 1). Compare the measurement value to the initial calibration value. This difference in value is then compared to the drift criteria or post calibration criteria described in the quality assurance project plan or the sampling and analysis plan for the project. If the check value is outside the criteria, then the measurement data will need to be qualified.

For the dissolved oxygen calibration check, follow the calibration instructions steps one through three while the instrument is in measurement mode. Record dissolved oxygen value (mg/L), temperature, and barometric pressure. Compare the measurement value to the Oxygen Solubility at Indicated Pressure chart attached to this SOP. The value should be within the criteria specified for the project. If measurement value drifted outside the criteria, the data will need to be qualified.

If the quality assurance project plan or the sampling and analysis plan do not list the drift criteria or the post-calibration criteria, use the criteria below.

Measurement	**Post Calibration Criteria**
Dissolved Oxygen	± 0.5 mg/L of sat. value* < 0.5 mg/L for the 0 mg/L solution, but not a negative value
Specific Conductance	±5% of standard or ± 10 µS/cm (whichever is greater)
pH	± 0.3 pH unit with pH 7 buffer*
Turbidity	± 5% of standard
ORP	± 10 mv*

Note: * Table 8.1, USEPA Region 1 *YSI 6-Series Sondes and Data Logger SOP*, January 30, 2007, revision 9.

7.0 DATA MANAGEMENT AND RECORDS MANAGEMENT

All calibration records must be documented in the project's log book or on a calibration log sheet. At a minimum, include the instrument manufacturer, model number, instrument identification number (when more than one instrument of the same model is used), the standards used to calibrate the instruments (including source), the calibration date, the instrument readings, the post calibration check, and the name of the person(s) who performed the calibration. An example of a calibration log sheet is shown in Table 1.

8.0 References

Standard Methods for the Examination of Water and Wastewater, 20th edition, 1998.

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983.

Turbidity - Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993.

USEPA Region 1 YSI 6-Series Sondes and Data Logger SOP, January 30, 2007, revision 9.

USGS Guidelines and Standard Procedures for Continuous Water-Quality Monitors: Station Operation, Record Computation, and Data Reporting, Techniques and Methods 1-D3.

TABLE 1
INSTRUMENT CALIBRATION LOG

Project Name_____ Date_____
Weather_____

Calibrated by_____ Instrument_____
Serial Number_____

Parameters	Morning Calibration	Morning Temperature	End of Day Calibration Check*	End of Day Temperature
Specific Conductance Standard #1				
Specific Conductance Standard #2				
pH (7)				
pH (4)				
pH (10)				
ORP Zobel solution				
Dissolved Oxygen 100% water saturated air mg/L				
Dissolved Oxygen Zero Dissolved Oxygen Solution mg/L				
Barometric Pressure mm Hg		NA		NA
Turbidity Standard #1				
Turbidity Standard #2				
Turbidity Standard #3				

* For each Parameter, chose one standard as your check standard. If possible, choose the one that is closest to the ambient measurement value.

Oxygen Solubility at Indicated Pressure

Temp.	Pressure (Hg)							
	760	755	750	745	740	735	730	mm
°C	29.92	29.72	29.53	29.33	29.13	28.94	28.74	in
0	14.57	14.47	14.38	14.28	14.18	14.09	13.99	mg/l
1	14.17	14.08	13.98	13.89	13.79	13.70	13.61	
2	13.79	13.70	13.61	13.52	13.42	13.33	13.24	
3	13.43	13.34	13.25	13.16	13.07	12-98	12.90	
4	13.08	12.99	12.91	12.82	12.73	12.65	12.56	
5	12.74	12.66	12.57	12.49	12.40	12.32	12.23	
6	12.42	12.34	12.26	12.17	12.09	12.01	11.93	
7	12.11	12.03	11.95	11.87	11.79	11.71	11.63	
8	11.81	11.73	11.65	11.57	11.50	11.42	11.34	
9	11.53	11.45	11.38	11.30	11.22	11.15	11.07	
10	11.28	11.19	11.11	11.04	10.96	10.89	10.81	
11	10.99	10.92	10.84	10.77	10.70	10.62	10.55	
12	10.74	10.67	10.60	10.53	10.45	10.38	10.31	
13	10.50	10.43	10.36	10.29	10.22	10.15	10.08	
14	10.27	10.20	10.13	10.06	10.00	9.93	9.86	
15	10.05	9.98	9.92	9.85	9.78	9.71	9.65	
16	9.83	9.76	9.70	9.63	9.57	9.50	9.43	
17	9.63	9.57	9.50	9.44	9.37	9.31	9.24	
18	9.43	9.37	9.30	9.24	9.18	9.11	9.05	
19	9.24	9.18	9.12	9.05	8.99	8.93	8.87	
20	9.06	9.00	8.94	8.88	8.82	8.75	8.69	
21	8.88	8.82	8.76	8.70	8.64	8.58	8.52	
22	8.71	8.65	8.59	8.53	8.47	8.42	8.36	
23	8.55	8.49	8.43	8.38	8.32	8.26	8.20	
24	8.39	8.33	8.28	8.22	8.16	8.11	8.05	
25	8.24	8.18	8.13	8.07	8.02	7.96	7.90	
26	8.09	8.03	7.98	7.92	7.87	7.81	7.76	
27	7.95	7.90	7.84	7.79	7.73	7.68	7.62	
28	7.81	7.76	7.70	7.65	7.60	7.54	7.49	
29	7.68	7.63	7.57	7.52	7.47	7.42	7.36	
30	7.55	7.50	7.45	7.39	7.34	7.29	7.24	
31	7.42	7.37	7.32	7.27	7.22	7.16	7.11	
32	7.30	7.25	7.20	7.15	7.10	7.05	7.00	
33	7.08	7.13	7.08	7.03	6.98	6.93	6.88	
34	7.07	7.02	6.97	6.92	6.87	6.82	6.78	
35	6.95	6.90	6.85	6.80	6.76	6.71	6.66	
36	6.84	6.79	6.76	6.70	6.65	6.60	6.55	
37	6.73	6.68	6.64	6.59	6.54	6.49	6.45	
38	6.63	6.58	6.54	6.49	6.44	6.40	6.35	
39	6.52	6.47	6.43	6.38	6.35	6.29	6.24	
40	6.42	6.37	6.33	6.28	6.24	6.19	6.15	
41	6.32	6.27	6.23	6.18	6.14	6.09	6.05	
42	6.22	6.18	6.13	6.09	6.04	6.00	5.95	
43	6.13	6.09	6.04	6.00	5.95	5.91	5.87	
44	6.03	5.99	5.94	5.90	5.86	5.81	5.77	
45	5.94	5.90	5.85	5.81	5.77	5.72	5.68	

(Continued)

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.

Oxygen Solubility at Indicated Pressure (continued)

Temp.	Pressure (Hg)								
	725	720	715	710	705	700	695	690	mm
°C	28.54	28.35	28.15	27.95	27.76	27.56	27.36	27.17	in
0	13.89	13.80	13.70	13.61	13.51	13.41	13.32	13.22	mg/l
1	13.51	13.42	13.33	13.23	13.14	13.04	12.95	12.86	
2	13.15	13.06	12.07	12.88	12.79	12.69	12.60	12.51	
3	12.81	12.72	12.63	12.54	12.45	12.36	12.27	12.18	
4	12.47	12.39	12.30	12.21	12.13	12.04	11.95	11.87	
5	12.15	12.06	11.98	11.89	11.81	11.73	11.64	11.56	
6	11.84	11.73	11.68	11.60	11.51	11.43	11.35	11.27	
7	11.55	11.47	11.39	11.31	11.22	11.14	11.06	10.98	
8	11.26	11.18	11.10	11.02	10.95	10.87	10.79	10.71	
9	10.99	10.92	10.84	10.76	10.69	10.61	10.53	10.46	
10	10.74	10.66	10.59	10.51	10.44	10.36	10.29	10.21	
11	10.48	10.40	10.33	10.28	10.18	10.11	10.04	9.96	
12	10.24	10.17	10.10	10.02	9.95	9.88	9.81	9.46	
13	10.01	9.94	9.87	9.80	9.73	9.66	9.59	9.52	
14	9.79	9.72	9.65	9.68	9.51	9.45	9.38	9.31	
15	9.58	9.51	9.44	9.58	9.31	9.24	9.18	9.11	
16	9.37	9.30	9.24	9.17	9.11	9.04	8.97	8.91	
17	9.18	9.11	9.05	8.98	8.92	8.85	8.79	8.73	
18	8.99	8.92	8.86	8.80	8.73	8.67	8.61	8.54	
19	8.81	8.74	8.68	8.62	8.56	8.49	8.43	8.37	
20	8.63	8.57	8.51	8.45	8.39	8.33	8.27	8.21	
21	8.46	8.40	8.34	8.28	8.22	8.16	8.10	8.04	
22	8.30	8.24	8.18	8.12	8.06	8.00	7.95	7.89	
23	8.15	8.09	8.03	7.97	7.91	7.86	7.80	7.74	
24	7.99	7.94	7.88	7.82	7.76	7.71	7.65	7.59	
25	7.85	7.79	7.74	7.68	7.60	7.57	7.51	7.46	
26	7.70	7.65	7.59	7.54	7.48	7.43	7.37	7.32	
27	7.57	7.52	7.46	7.41	7.35	7.30	7.25	7.19	
28	7.44	7.38	7.33	7.28	7.22	7.17	7.12	7.06	
29	7.31	7.26	7.21	7.15	7.10	7.05	7.00	6.94	
30	7.19	7.14	7.08	7.03	6.98	6.93	6.88	6.82	
31	7.06	7.01	6.96	6.91	6.86	6.81	6.76	6.70	
32	6.95	6.90	6.85	6.80	6.70	6.70	6.64	6.59	
33	6.83	6.78	6.73	6.68	6.83	6.58	6.53	6.48	
34	6.73	6.68	6.63	6.58	6.53	6.48	6.43	6.38	
35	6.61	6.56	6.51	6.47	6.42	6.37	6.36	6.27	
36	6.51	6.46	6.41	6.36	6.31	6.27	6.22	6.17	
37	6.40	6.35	6.31	6.26	6.21	6.16	6.12	6.07	
38	6.30	6.26	6.21	6.16	6.12	6.07	6.02	5.98	
39	6.26	6.15	6.11	6.06	6.01	5.97	5.92	5.87	
40	6.10	6.06	6.01	5.96	5.92	5.86	5.83	5.78	
41	6.00	5.96	5.91	5.87	5.82	5.78	5.73	5.69	
42	5.91	5.86	5.82	5.77	5.73	5.69	5.64	5.60	
43	5.82	5.78	5.73	5.69	5.65	5.60	5.56	5.51	
44	5.72	5.68	5.64	5.59	5.55	5.51	5.46	5.42	
45	5.64	5.59	5.55	5.51	5.47	5.42	5.38	5.34	

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUNDWATER SAMPLES FROM MONITORING WELLS

Quality Assurance Unit
U.S. Environmental Protection Agency – Region 1
11 Technology Drive
North Chelmsford, MA 01863

The controlled version of this document is the electronic version viewed on-line only. If this is a printed copy of the document, it is an uncontrolled version and may or may not be the version currently in use.

This document contains direction developed solely to provide guidance to U.S. Environmental Protection Agency (EPA) personnel. EPA retains the discretion to adopt approaches that differ from these procedures on a case-by-case basis. The procedures set forth do not create any rights, substantive or procedural, enforceable at law by party to litigation with EPA or the United States.

Prepared by: Charles Porfert 1/19/10
(Charles Porfert, Quality Assurance Unit) Date

Approved by: Gerard Sotolongo 1-19-10
(Gerard Sotolongo, Quality Assurance Unit) Date

Revision Page

Date	Rev #	Summary of changes	Sections
7/30/96	2	Finalized	
01/19/10	3	Updated	All sections

TABLE OF CONTENTS

	Page
USE OF TERMS	4
SCOPE & APPLICATION	5
BACKGROUND FOR IMPLEMENTATION	6
HEALTH & SAFETY	7
CAUTIONS	7
PERSONNEL QUALIFICATIONS	9
EQUIPMENT AND SUPPLIES	9
EQUIPMENT/INSTRUMENT CALIBRATION	13
PRELIMINARY SITE ACTIVITIES	13
PURGING AND SAMPLING PROCEDURE	14
DECONTAMINATION	19
FIELD QUALITY CONTROL	21
FIELD LOGBOOK	21
DATA REPORT	22
REFERENCES	22
APPENDIX A PERISTALTIC PUMPS	24
APPENDIX B SUMMARY OF SAMPLING INSTRUCTIONS	25
LOW-FLOW SETUP DIAGRAM	29
APPENDIX C EXAMPLE WELL PURGING FORM	30

USE OF TERMS

Equipment blank: The equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank needs only to include the pump in subsequent sampling rounds. If the pump and tubing are dedicated to the well, the equipment blank is collected prior to its placement in the well. If the pump and tubing will be used to sample multiple wells, the equipment blank is normally collected after sampling from contaminated wells and not after background wells.

Field duplicates: Field duplicates are collected to determine precision of the sampling procedure. For this procedure, collect duplicate for each analyte group in consecutive order (VOC original, VOC duplicate, SVOC original, SVOC duplicate, etc.).

Indicator field parameters: This SOP uses field measurements of turbidity, dissolved oxygen, specific conductance, temperature, pH, and oxidation/reduction potential (ORP) as indicators of when purging operations are sufficient and sample collection may begin.

Matrix Spike/Matrix Spike Duplicates: Used by the laboratory in its quality assurance program. Consult the laboratory for the sample volume to be collected.

Poteniometric Surface: The level to which water rises in a tightly cased well constructed in a confined aquifer. In an unconfined aquifer, the potentiometric surface is the water table.

QAPP: Quality Assurance Project Plan

SAP: Sampling and Analysis Plan

SOP: Standard operating procedure

Stabilization: A condition that is achieved when all indicator field parameter measurements are sufficiently stable (as described in the "Monitoring Indicator Field Parameters" section) to allow sample collection to begin.

Temperature blank: A temperature blank is added to each sample cooler. The blank is measured upon receipt at the laboratory to assess whether the samples were properly cooled during transit.

Trip blank (VOCs): Trip blank is a sample of analyte-free water taken to the sampling site and returned to the laboratory. The trip blanks (one pair) are added to each sample cooler that contains VOC samples.

SCOPE & APPLICATION

The goal of this groundwater sampling procedure is to collect water samples that reflect the total mobile organic and inorganic loads (dissolved and colloidal sized fractions) transported through the subsurface under ambient flow conditions, with minimal physical and chemical alterations from sampling operations. This standard operating procedure (SOP) for collecting groundwater samples will help ensure that the project's data quality objectives (DQOs) are met under certain low-flow conditions.

The SOP emphasizes the need to minimize hydraulic stress at the well-aquifer interface by maintaining low water-level drawdowns, and by using low pumping rates during purging and sampling operations. Indicator field parameters (e.g., dissolved oxygen, pH, etc.) are monitored during purging in order to determine when sample collection may begin. Samples properly collected using this SOP are suitable for analysis of groundwater contaminants (volatile and semi-volatile organic analytes, dissolved gases, pesticides, PCBs, metals and other inorganics), or naturally occurring analytes. This SOP is based on Puls, and Barcelona (1996).

This procedure is designed for monitoring wells with an inside diameter (1.5-inches or greater) that can accommodate a positive lift pump with a screen length or open interval ten feet or less and with a water level above the top of the screen or open interval (Hereafter, the "screen or open interval" will be referred to only as "screen interval"). This SOP is not applicable to other well-sampling conditions.

While the use of dedicated sampling equipment is not mandatory, dedicated pumps and tubing can reduce sampling costs significantly by streamlining sampling activities and thereby reducing the overall field costs.

The goal of this procedure is to emphasize the need for consistency in deploying and operating equipment while purging and sampling monitoring wells during each sampling event. This will help to minimize sampling variability.

This procedure describes a general framework for groundwater sampling. Other site specific information (hydrogeological context, conceptual site model (CSM), DQOs, etc.) coupled with systematic planning must be added to the procedure in order to develop an appropriate site specific SAP/QAPP. In addition, the site specific SAP/QAPP must identify the specific equipment that will be used to collect the groundwater samples.

This procedure does not address the collection of water or free product samples from wells containing free phase LNAPLs and/or DNAPLs (light or dense non-aqueous phase

liquids). For this type of situation, the reader may wish to check: Cohen, and Mercer (1993) or other pertinent documents.

This SOP is to be used when collecting groundwater samples from monitoring wells at all Superfund, Federal Facility and RCRA sites in Region 1 under the conditions described herein. Request for modification of this SOP, in order to better address specific situations at individual wells, must include adequate technical justification for proposed changes. All changes and modifications must be approved and included in a revised SAP/QAPP before implementation in field.

BACKGROUND FOR IMPLEMENTATION

It is expected that the monitoring well screen has been properly located (both laterally and vertically) to intercept existing contaminant plume(s) or along flow paths of potential contaminant migration. Problems with inappropriate monitoring well placement or faulty/improper well installation cannot be overcome by even the best water sampling procedures. This SOP presumes that the analytes of interest are moving (or will potentially move) primarily through the more permeable zones intercepted by the screen interval.

Proper well construction, development, and operation and maintenance cannot be overemphasized. The use of installation techniques that are appropriate to the hydrogeologic setting of the site often prevent "problem well" situations from occurring. During well development, or redevelopment, tests should be conducted to determine the hydraulic characteristics of the monitoring well. The data can then be used to set the purging/sampling rate, and provide a baseline for evaluating changes in well performance and the potential need for well rehabilitation. Note: if this installation data or well history (construction and sampling) is not available or discoverable, for all wells to be sampled, efforts to build a sampling history should commence with the next sampling event.

The pump intake should be located within the screen interval and at a depth that will remain under water at all times. It is recommended that the intake depth and pumping rate remain the same for all sampling events. The mid-point or the lowest historical midpoint of the saturated screen length is often used as the location of the pump intake. For new wells, or for wells without pump intake depth information, the site's SAP/QAPP must provide clear reasons and instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected. If the depths to top and bottom of the well screen are not known, the SAP/QAPP will need to describe how the sampling depth will be determined and how the data can be used.

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU, and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. Sample collection

may still take place provided the indicator field parameter criteria in this procedure are met. If after 2 hours of purging indicator field parameters have not stabilized, one of three optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization), c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may reflect a sampling bias and therefore, the data may not meet the data quality objectives of the sampling event).

It is recommended that low-flow sampling be conducted when the air temperature is above 32°F (0°C). If the procedure is used below 32°F, special precautions will need to be taken to prevent the groundwater from freezing in the equipment. Because sampling during freezing temperatures may adversely impact the data quality objectives, the need for water sample collection during months when these conditions are likely to occur should be evaluated during site planning and special sampling measures may need to be developed. Ice formation in the flow-through-cell will cause the monitoring probes to act erratically. A transparent flow-through-cell needs to be used to observe if ice is forming in the cell. If ice starts to form on the other pieces of the sampling equipment, additional problems may occur.

HEALTH & SAFETY

When working on-site, comply with all applicable OSHA requirements and the site's health/safety procedures. All proper personal protection clothing and equipment are to be worn. Some samples may contain biological and chemical hazards. These samples should be handled with suitable protection to skin, eyes, etc.

CAUTIONS

The following cautions need to be considered when planning to collect groundwater samples when the below conditions occur.

If the groundwater degasses during purging of the monitoring well, dissolved gases and VOCs will be lost. When this happens, the groundwater data for dissolved gases (e.g., methane, ethene, ethane, dissolved oxygen, etc.) and VOCs will need to be qualified. Some conditions that can promote degassing are the use of a vacuum pump (e.g., peristaltic pumps), changes in aperture along the sampling tubing, and squeezing/pinching the pump's tubing which results in a pressure change.

When collecting the samples for dissolved gases and VOCs analyses, avoid aerating the groundwater in the pump's tubing. This can cause loss of the dissolved gases and VOCs in

the groundwater. Having the pump's tubing completely filled prior to sampling will avoid this problem when using a centrifugal pump or peristaltic pump.

Direct sun light and hot ambient air temperatures may cause the groundwater in the tubing and flow-through-cell to heat up. This may cause the groundwater to degas which will result in loss of VOCs and dissolved gases. When sampling under these conditions, the sampler will need to shade the equipment from the sunlight (e.g., umbrella, tent, etc.). If possible, sampling on hot days, or during the hottest time of the day, should be avoided. The tubing exiting the monitoring well should be kept as short as possible to avoid the sun light or ambient air from heating up the groundwater.

Thermal currents in the monitoring well may cause vertical mixing of water in the well bore. When the air temperature is colder than the groundwater temperature, it can cool the top of the water column. Colder water which is denser than warm water sinks to the bottom of the well and the warmer water at the bottom of the well rises, setting up a convention cell. "During low-flow sampling, the pumped water may be a mixture of convecting water from within the well casing and aquifer water moving inward through the screen. This mixing of water during low-flow sampling can substantially increase equilibration times, can cause false stabilization of indicator parameters, can give false indication of redox state, and can provide biological data that are not representative of the aquifer conditions" (Vroblesky 2007).

Failure to calibrate or perform proper maintenance on the sampling equipment and measurement instruments (e.g., dissolved oxygen meter, etc.) can result in faulty data being collected.

Interferences may result from using contaminated equipment, cleaning materials, sample containers, or uncontrolled ambient/surrounding air conditions (e.g., truck/vehicle exhaust nearby).

Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and/or proper planning to avoid ambient air interferences. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

Clean and decontaminate all sampling equipment prior to use. All sampling equipment needs to be routinely checked to be free from contaminants and equipment blanks collected to ensure that the equipment is free of contaminants. Check the previous equipment blank data for the site (if they exist) to determine if the previous cleaning procedure removed the contaminants. If contaminants were detected and they are a concern, then a more vigorous cleaning procedure will be needed.

PERSONNEL QUALIFICATIONS

All field samplers working at sites containing hazardous waste must meet the requirements of the OSHA regulations. OSHA regulations may require the sampler to take the 40 hour OSHA health and safety training course and a refresher course prior to engaging in any field activities, depending upon the site and field conditions.

The field samplers must be trained prior to the use of the sampling equipment, field instruments, and procedures. Training is to be conducted by an experienced sampler before initiating any sampling procedure.

The entire sampling team needs to read, and be familiar with, the site Health and Safety Plan, all relevant SOPs, and SAP/QAPP (and the most recent amendments) before going onsite for the sampling event. It is recommended that the field sampling leader attest to the understanding of these site documents and that it is recorded.

EQUIPMENT AND SUPPLIES

A. Informational materials for sampling event

A copy of the current Health and Safety Plan, SAP/QAPP, monitoring well construction data, location map(s), field data from last sampling event, manuals for sampling, and the monitoring instruments' operation, maintenance, and calibration manuals should be brought to the site.

B. Well keys.

C. Extraction device

Adjustable rate, submersible pumps (e.g., centrifugal, bladder, etc.) which are constructed of stainless steel or Teflon are preferred. Note: if extraction devices constructed of other materials are to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

If bladder pumps are selected for the collection of VOCs and dissolved gases, the pump setting should be set so that one pulse will deliver a water volume that is sufficient to fill a 40 mL VOC vial. This is not mandatory, but is considered a "best practice". For the proper operation, the bladder pump will need a minimum amount of water above the pump; consult the manufacturer for the recommended submergence. The pump's recommended submergence value should be determined during the planning stage, since it may influence well construction and placement of dedicated pumps where water-level fluctuations are significant.

Adjustable rate, peristaltic pumps (suction) are to be used with caution when collecting samples for VOCs and dissolved gases (e.g., methane, carbon dioxide, etc.) analyses. Additional information on the use of peristaltic pumps can be found in Appendix A. If peristaltic pumps are used, the inside diameter of the rotor head tubing needs to match the inside diameter of the tubing installed in the monitoring well.

Inertial pumping devices (motor driven or manual) are not recommended. These devices frequently cause greater disturbance during purging and sampling, and are less easily controlled than submersible pumps (potentially increasing turbidity and sampling variability, etc.). This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

D. Tubing

Teflon or Teflon-lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics. Note: if tubing constructed of other materials is to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for metal and other inorganics analyses.

The use of 1/4 inch or 3/8 inch (inside diameter) tubing is recommended. This will help ensure that the tubing remains liquid filled when operating at very low pumping rates when using centrifugal and peristaltic pumps.

Silastic tubing should be used for the section around the rotor head of a peristaltic pump. It should be less than a foot in length. The inside diameter of the tubing used at the pump rotor head must be the same as the inside diameter of tubing placed in the well. A tubing connector is used to connect the pump rotor head tubing to the well tubing. Alternatively, the two pieces of tubing can be connected to each other by placing the one end of the tubing inside the end of the other tubing. The tubing must not be reused.

E. The water level measuring device

Electronic "tape", pressure transducer, water level sounder/level indicator, etc. should be capable of measuring to 0.01 foot accuracy. Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each sampling event.

F. Flow measurement supplies

Graduated cylinder (size according to flow rate) and stopwatch usually will suffice.

Large graduated bucket used to record total water purged from the well.

G. Interface probe

To be used to check on the presence of free phase liquids (LNAPL, or DNAPL) before purging begins (as needed).

H. Power source (generator, nitrogen tank, battery, etc.)

When a gasoline generator is used, locate it downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate samples.

I. Indicator field parameter monitoring instruments

Use of a multi-parameter instrument capable of measuring pH, oxidation/reduction potential (ORP), dissolved oxygen (DO), specific conductance, temperature, and coupled with a flow-through-cell is required when measuring all indicator field parameters, except turbidity. Turbidity is collected using a separate instrument. Record equipment/instrument identification (manufacturer, and model number).

Transparent, small volume flow-through-cells (e.g., 250 mLs or less) are preferred. This allows observation of air bubbles and sediment buildup in the cell, which can interfere with the operation of the monitoring instrument probes, to be easily detected. A small volume cell facilitates rapid turnover of water in the cell between measurements of the indicator field parameters.

It is recommended to use a flow-through-cell and monitoring probes from the same manufacturer and model to avoid incompatibility between the probes and flow-through-cell.

Turbidity samples are collected before the flow-through-cell. A "T" connector coupled with a valve is connected between the pump's tubing and flow-through-cell. When a turbidity measurement is required, the valve is opened to allow the groundwater to flow into a container. The valve is closed and the container sample is then placed in the turbidimeter.

Standards are necessary to perform field calibration of instruments. A minimum of two standards are needed to bracket the instrument measurement range for all parameters except ORP which use a Zobell solution as a standard. For dissolved oxygen, a wet sponge used for the 100% saturation and a zero dissolved oxygen solution are used for the calibration.

Barometer (used in the calibration of the Dissolved Oxygen probe) and the conversion formula to convert the barometric pressure into the units of measure used by the Dissolved Oxygen meter are needed.

J. Decontamination supplies

Includes (for example) non-phosphate detergent, distilled/deionized water, isopropyl alcohol, etc.

K. Record keeping supplies

Logbook(s), well purging forms, chain-of-custody forms, field instrument calibration forms, etc.

L. Sample bottles

M. Sample preservation supplies (as required by the analytical methods)

N. Sample tags or labels

O. PID or FID instrument

If appropriate, to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

P. Miscellaneous Equipment

Equipment to keep the sampling apparatus shaded in the summer (e.g., umbrella) and from freezing in the winter. If the pump's tubing is allowed to heat up in the warm weather, the cold groundwater may degas as it is warmed in the tubing.

EQUIPMENT/INSTRUMENT CALIBRATION

Prior to the sampling event, perform maintenance checks on the equipment and instruments according to the manufacturer's manual and/or applicable SOP. This will ensure that the equipment/instruments are working properly before they are used in the field.

Prior to sampling, the monitoring instruments must be calibrated and the calibration documented. The instruments are calibrated using U.S Environmental Protection Agency Region 1 *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity),* January 19, 2010, or latest version or from one of the methods listed in 40CFR136, 40CFR141 and SW-846.

The instruments shall be calibrated at the beginning of each day. If the field measurement falls outside the calibration range, the instrument must be re-calibrated so that all measurements fall within the calibration range. At the end of each day, a calibration check is performed to verify that instruments remained in calibration throughout the day. This check is performed while the instrument is in measurement mode, not calibration mode. If the field instruments are being used to monitor the natural attenuation parameters, then a calibration check at mid-day is highly recommended to ensure that the instruments did not drift out of calibration. Note: during the day if the instrument reads zero or a negative number for dissolved oxygen, pH, specific conductance, or turbidity (negative value only), this indicates that the instrument drifted out of calibration or the instrument is malfunctioning. If this situation occurs the data from this instrument will need to be qualified or rejected.

PRELIMINARY SITE ACTIVITIES (as applicable)

Check the well for security (damage, evidence of tampering, missing lock, etc.) and record pertinent observations (include photograph as warranted).

If needed lay out sheet of clean polyethylene for monitoring and sampling equipment, unless equipment is elevated above the ground (e.g., on a table, etc.).

Remove well cap and if appropriate measure VOCs at the rim of the well with a PID or FID instrument and record reading in field logbook or on the well purge form.

If the well casing does not have an established reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook (consider a photographic record as well). All water level measurements must be recorded relative to this reference point (and the altitude of this point should be determined using techniques that are appropriate to site's DQOs.

If water-table or potentiometric surface map(s) are to be constructed for the sampling event, perform synoptic water level measurement round (in the shortest possible time) before any purging and sampling activities begin. If possible, measure water level depth (to 0.01 ft.) and total well depth (to 0.1 ft.) the day before sampling begins, in order to allow for re-settlement of any particulates in the water column. This is especially important for those wells that have not been recently sampled because sediment buildup in the well may require the well to be redeveloped. If measurement of total well depth is not made the day before, it should be measured after sampling of the well is complete. All measurements must be taken from the established referenced point. Care should be taken to minimize water column disturbance.

Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent check measurements with an interface probe may not be necessary unless analytical data or field analysis signal a worsening situation. This SOP cannot be used in the presence of LNAPLs or DNAPLs. If NAPLs are present, the project team must decide upon an alternate sampling method. All project modifications must be approved and documented prior to implementation.

If available check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use final pump dial setting information) from previous event(s). If changes are made in the intake depth or extraction rate(s) used during previous sampling event(s), for either portable or dedicated extraction devices, record new values, and explain reasons for the changes in the field logbook.

PURGING AND SAMPLING PROCEDURE

Purging and sampling wells in order of increasing chemical concentrations (known or anticipated) are preferred.

The use of dedicated pumps is recommended to minimize artificial mobilization and entrainment of particulates each time the well is sampled. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each

sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

A. Initial Water Level

Measure the water level in the well before installing the pump if a non-dedicated pump is being used. The initial water level is recorded on the purge form or in the field logbook.

B. Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the appropriate depth (may not be the mid-point of the screen/open interval). The Sampling and Analysis Plan/Quality Assurance Project Plan should specify the sampling depth (used previously), or provide criteria for selection of intake depth for each new well. If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well.

Pump tubing lengths, above the top of well casing should be kept as short as possible to minimize heating the groundwater in the tubing by exposure to sun light and ambient air temperatures. Heating may cause the groundwater to degas, which is unacceptable for the collection of samples for VOC and dissolved gases analyses.

C. Measure Water Level

Before starting pump, measure water level. Install recording pressure transducer, if used to track drawdowns, to initialize starting condition.

D. Purge Well

From the time the pump starts purging and until the time the samples are collected, the purged water is discharged into a graduated bucket to determine the total volume of groundwater purged. This information is recorded on the purge form or in the field logbook.

Start the pump at low speed and slowly increase the speed until discharge occurs. Check water level. Check equipment for water leaks and if present fix or replace the affected equipment. Try to match pumping rate used during previous sampling event(s). Otherwise, adjust pump speed until there is little or no water level drawdown. If the minimal drawdown that can be achieved exceeds 0.3 feet, but remains stable, continue purging.

Monitor and record the water level and pumping rate every five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" somewhat as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. If the initial water level is above the top of the screen do not allow the water level to fall into the well screen. The final purge volume must be greater than the stabilized drawdown volume plus the pump's tubing volume. If the drawdown has exceeded 0.3 feet and stabilizes, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are collected.

Avoid the use of constriction devices on the tubing to decrease the flow rate because the constrictor will cause a pressure difference in the water column. This will cause the groundwater to degas and result in a loss of VOCs and dissolved gasses in the groundwater samples.

Note: the flow rate used to achieve a stable pumping level should remain constant while monitoring the indicator parameters for stabilization and while collecting the samples.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (e.g., bladder, peristaltic), and/or the use of dedicated equipment. For new monitoring wells, or wells where the following situation has not occurred before, if the recovery rate to the well is less than 50 mL/min., or the well is being essentially dewatered during purging, the well should be sampled as soon as the water level has recovered sufficiently to collect the volume needed for all anticipated samples. The project manager or field team leader will need to make the decision when samples should be collected, how the sample is to be collected, and the reasons recorded on the purge form or in the field logbook. A water level measurement needs to be performed and recorded before samples are collected. If the project manager decides to collect the samples using the pump, it is best during this recovery period that the pump intake tubing not be removed, since this will aggravate any turbidity problems. Samples in this specific situation may be collected without stabilization of indicator field parameters. Note that field conditions and efforts to overcome problematic situations must be recorded in order to support field decisions to deviate from normal procedures described in this SOP. If this type of problematic situation persists in a well, then water sample collection should be changed to a passive or no-purge method, if consistent with the site's DQOs, or have a new well installed.

E. Monitor Indicator Field Parameters

After the water level has stabilized, connect the "T" connector with a valve and the flow-through-cell to monitor the indicator field parameters. If excessive turbidity is anticipated or encountered with the pump startup, the well may be purged for a while without connecting up the flow-through-cell, in order to minimize particulate buildup in the cell (This is a judgment call made by the sampler). Water level drawdown measurements should be made as usual. If possible, the pump may be installed the day before purging to allow particulates that were disturbed during pump insertion to settle.

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, ORP, DO) at a frequency of five minute intervals or greater. The pump's flow rate must be able to "turn over" at least one flow-through-cell volume between measurements (for a 250 mL flow-through-cell with a flow rate of 50 mLs/min., the monitoring frequency would be every five minutes; for a 500 mL flow-through-cell it would be every ten minutes). If the cell volume cannot be replaced in the five minute interval, then the time between measurements must be increased accordingly. Note: during the early phase of purging emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments followed by stabilization of indicator parameters. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings are within the following limits:

Turbidity (10% for values greater than 5 NTU; if three Turbidity values are less than 5 NTU, consider the values as stabilized),
Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),
Specific Conductance (3%),
Temperature (3%),
pH (± 0.1 unit),
Oxidation/Reduction Potential (±10 millivolts).

All measurements, except turbidity, must be obtained using a flow-through-cell. Samples for turbidity measurements are obtained before water enters the flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values measured within the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities. Record start and stop times and give a brief description of cleaning activities.

The flow-through-cell must be designed in a way that prevents gas bubble entrapment in the cell. Placing the flow-through-cell at a 45 degree angle with the port facing upward can help remove bubbles from the flow-through-cell (see Appendix B Low-Flow Setup Diagram). All during the measurement process, the flow-through-cell must remain free of any gas bubbles. Otherwise, the monitoring probes may act erratically. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must remain submerged in water at all times.

F. Collect Water Samples

When samples are collected for laboratory analyses, the pump's tubing is disconnected from the "T" connector with a valve and the flow-through-cell. The samples are collected directly from the pump's tubing. Samples must not be collected from the flow-through-cell or from the "T" connector with a valve.

VOC samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the pump's flow rate is too high to collect the VOC/dissolved gases samples, collect the other samples first. Lower the pump's flow rate to a reasonable rate and collect the VOC/dissolved gases samples and record the new flow rate.

During purging and sampling, the centrifugal/peristaltic pump tubing must remain filled with water to avoid aeration of the groundwater. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help insure that the sample tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use the following procedure to collect samples: collect non-VOC/dissolved gases samples first, then increase flow rate slightly until the water completely fills the tubing, collect the VOC/dissolved gases samples, and record new drawdown depth and flow rate.

For bladder pumps that will be used to collect VOC or dissolved gas samples, it is recommended that the pump be set to deliver long pulses of water so that one pulse will fill a 40 mL VOC vial.

Use pre-preserved sample containers or add preservative, as required by analytical methods, to the samples immediately after they are collected. Check the analytical methods (e.g. EPA SW-846, 40 CFR 136, water supply, etc.) for additional information on preservation.

If determination of filtered metal concentrations is a sampling objective, collect filtered water samples using the same low flow procedures. The use of an in-line filter (transparent housing preferred) is required, and the filter size (0.45 μm is commonly used) should be based on the sampling objective. Pre-rinse the filter with groundwater prior to sample collection. Make sure the filter is free of air bubbles before samples are collected. Preserve the filtered water sample immediately. Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in groundwater for human health or ecological risk calculations.

Label each sample as collected. Samples requiring cooling will be placed into a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

G. Post Sampling Activities

If a recording pressure transducer is used to track drawdown, re-measure water level with tape.

After collection of samples, the pump tubing may be dedicated to the well for re-sampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.1 ft.), if not measured the day before purging began. Note: measurement of total well depth annually is usually sufficient after the initial low stress sampling event. However, a greater frequency may be needed if the well has a "silting" problem or if confirmation of well identity is needed.

Secure the well.

DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well and then following sampling of each well. Pumps should not be removed between purging and sampling operations. The pump, tubing, support cable and electrical wires which were in contact with the well should be decontaminated by one of the procedures listed below.

The use of dedicated pumps and tubing will reduce the amount of time spent on decontamination of the equipment. If dedicated pumps and tubing are used, only the initial sampling event will require decontamination of the pump and tubing.

Note if the previous equipment blank data showed that contaminant(s) were present after using the below procedure or the one described in the SAP/QAPP, a more vigorous procedure may be needed.

Procedure 1

Decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump and tubing. The pump may be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior and electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

Flush the equipment/pump with potable water.

Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.

Flush with potable or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.

Optional - flush with isopropyl alcohol (pesticide grade; must be free of ketones {e.g., acetone}) or with methanol. This step may be required if the well is highly contaminated or if the equipment blank data from the previous sampling event show that the level of contaminants is significant.

Flush with distilled/deionized water. This step must remove all traces of alcohol (if used) from the equipment. The final water rinse must not be recycled.

Procedure 2

Steam clean the outside of the submersible pump.

Pump hot potable water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three or four inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. The hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.

Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.

Pump potable water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.

Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the groundwater samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. Quality control samples include field duplicates, equipment blanks, matrix spike/matrix spike duplicates, trip blanks (VOCs), and temperature blanks.

FIELD LOGBOOK

A field log shall be kept to document all groundwater field monitoring activities (see Appendix C, example table), and record the following for each well:

Site name, municipality, state.

Well identifier, latitude-longitude or state grid coordinates.

Measuring point description (e.g., north side of PVC pipe).

Well depth, and measurement technique.

Well screen length.

Pump depth.

Static water level depth, date, time and measurement technique.

Presence and thickness of immiscible liquid (NAPL) layers and detection method.

Pumping rate, drawdown, indicator parameters values, calculated or measured total volume pumped, and clock time of each set of measurements.

Type of tubing used and its length.

Type of pump used.

Clock time of start and end of purging and sampling activity.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analyses.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions, including approximate ambient air temperature.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

Description of all sampling/monitoring equipment used, including trade names, model number, instrument identification number, diameters, material composition, etc.

DATA REPORT

Data reports are to include laboratory analytical results, QA/QC information, field indicator parameters measured during purging, field instrument calibration information, and whatever other field logbook information is needed to allow for a full evaluation of data usability.

Note: the use of trade, product, or firm names in this sampling procedure is for descriptive purposes only and does not constitute endorsement by the U.S. EPA.

REFERENCES

Cohen, R.M. and J.W. Mercer, 1993, *DNAPL Site Evaluation*; C.K. Smoley (CRC Press), Boca Raton, Florida.

Robert W. Puls and Michael J. Barcelona, *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*, April 1996 (EPA/540/S-95/504).

U.S. Environmental Protection Agency, 1992, *RCRA Ground-Water Monitoring: Draft Technical Guidance*; Washington, DC (EPA/530-R-93-001).

U.S. Environmental Protection Agency, 1987, *A Compendium of Superfund Field Operations Methods*; Washington, DC (EPA/540/P-87/001).

U.S Environmental Protection Agency, Region 1, *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity),* January 19, 2010 or latest version.

U.S Environmental Protection Agency, EPA SW-846.

U.S Environmental Protection Agency, 40 CFR 136.

U.S Environmental Protection Agency, 40 CFR 141.

Vroblesky, Don A., Clifton C. Casey, and Mark A. Lowery, Summer 2007, Influence of Dissolved Oxygen Convection on Well Sampling, *Ground Water Monitoring & Remediation* 27, no. 3: 49-58.

APPENDIX A
PERISTALTIC PUMPS

Before selecting a peristaltic pump to collect groundwater samples for VOCs and/or dissolved gases (e.g., methane, carbon dioxide, etc.) consideration should be given to the following:

- The decision of whether or not to use a peristaltic pump is dependent on the intended use of the data.
- If the additional sampling error that may be introduced by this device is NOT of concern for the VOC/dissolved gases data's intended use, then this device may be acceptable.
- If minor differences in the groundwater concentrations could effect the decision, such as to continue or terminate groundwater cleanup or whether the cleanup goals have been reached, then this device should NOT be used for VOC/dissolved gases sampling. In these cases, centrifugal or bladder pumps are a better choice for more accurate results.

EPA and USGS have documented their concerns with the use of the peristaltic pumps to collect water sample in the below documents.

- "Suction Pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987.
- "The agency does not recommend the use of peristaltic pumps to sample ground water particularly for volatile organic analytes" *RCRA Ground-Water Monitoring Draft Technical Guidance*, EPA Office of Solid Waste, November 1992.
- "The peristaltic pump is limited to shallow applications and can cause degassing resulting in alteration of pH, alkalinity, and volatiles loss", *Low-flow (Minimal drawdown) Ground-Water Sampling Procedures*, by Robert Puls & Michael Barcelona, April 1996, EPA/540/S-95/504.
- "Suction-lift pumps, such as peristaltic pumps, can operate at a very low pumping rate; however, using negative pressure to lift the sample can result in the loss of volatile analytes", USGS Book 9 Techniques of Water-Resources Investigation, Chapter A4. (Version 2.0, 9/2006).

APPENDIX B

SUMMARY OF SAMPLING INSTRUCTIONS

These instructions are for using an adjustable rate, submersible pump or a peristaltic pump with the pump's intake placed at the midpoint of a 10 foot or less well screen or an open interval. The water level in the monitoring well is above the top of the well screen or open interval, the ambient temperature is above 32°F, and the equipment is not dedicated. Field instruments are already calibrated. The equipment is setup according to the diagram at the end of these instructions.

1. Review well installation information. Record well depth, length of screen or open interval, and depth to top of the well screen. Determine the pump's intake depth (e.g., mid-point of screen/open interval).

2. On the day of sampling, check security of the well casing, perform any safety checks needed for the site, lay out a sheet of polyethylene around the well (if necessary), and setup the equipment. If necessary a canopy or an equivalent item can be setup to shade the pump's tubing and flow-through-cell from the sun light to prevent the sun light from heating the groundwater.

3. Check well casing for a reference mark. If missing, make a reference mark. Measure the water level (initial) to 0.01 ft. and record this information.

4. Install the pump's intake to the appropriate depth (e.g., midpoint) of the well screen or open interval. Do not turn-on the pump at this time.

5. Measure water level and record this information.

6. Turn-on the pump and discharge the groundwater into a graduated waste bucket. Slowly increase the flow rate until the water level starts to drop. Reduce the flow rate slightly so the water level stabilizes. Record the pump's settings. Calculate the flow rate using a graduated container and a stop watch. Record the flow rate. Do not let the water level drop below the top of the well screen.

If the groundwater is highly turbid or colored, continue to discharge the water into the bucket until the water clears (visual observation); this usually takes a few minutes. The turbid or colored water is usually from the well being disturbed during the pump installation. If the water does not clear, then you need to make a choice whether to continue purging the well (hoping that it will clear after a reasonable time) or continue to

the next step. Note, it is sometimes helpful to install the pump the day before the sampling event so that the disturbed materials in the well can settle out.

If the water level drops to the top of the well screen during the purging of the well, stop purging the well, and do the following:

> Wait for the well to recharge to a sufficient volume so samples can be collected. This may take awhile (pump maybe removed from well, if turbidity is not a problem). The project manager will need to make the decision when samples should be collected and the reasons recorded in the site's log book. A water level measurement needs to be performed and recorded before samples are collected. When samples are being collected, the water level must not drop below the top of the screen or open interval. Collect the samples from the pump's tubing. Always collect the VOCs and dissolved gases samples first. Normally, the samples requiring a small volume are collected before the large volume samples are collected just in case there is not sufficient water in the well to fill all the sample containers. All samples must be collected, preserved, and stored according to the analytical method. Remove the pump from the well and decontaminate the sampling equipment.

If the water level has dropped 0.3 feet or less from the initial water level (water level measure before the pump was installed); proceed to Step 7. If the water level has dropped more than 0.3 feet, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are be collected.

7. Attach the pump's tubing to the "T" connector with a valve (or a three-way stop cock). The pump's tubing from the well casing to the "T" connector must be as short as possible to prevent the groundwater in the tubing from heating up from the sun light or from the ambient air. Attach a short piece of tubing to the other end of the end of the "T" connector to serve as a sampling port for the turbidity samples. Attach the remaining end of the "T" connector to a short piece of tubing and connect the tubing to the flow-through-cell bottom port. To the top port, attach a small piece of tubing to direct the water into a calibrated waste bucket. Fill the cell with the groundwater and remove all gas bubbles from the cell. Position the flow-through-cell in such a way that if gas bubbles enter the cell they can easily exit the cell. If the ports are on the same side of the cell and the cell is cylindrical shape, the cell can be placed at a 45-degree angle with the ports facing upwards; this position should keep any gas bubbles entering the cell away from the monitoring probes and allow the gas bubbles to exit the cell easily (see Low-Flow Setup Diagram). Note,

make sure there are no gas bubbles caught in the probes' protective guard; you may need to shake the cell to remove these bubbles.

8. Turn-on the monitoring probes and turbidity meter.

9. Record the temperature, pH, dissolved oxygen, specific conductance, and oxidation/reduction potential measurements. Open the valve on the "T" connector to collect a sample for the turbidity measurement, close the valve, do the measurement, and record this measurement. Calculate the pump's flow rate from the water exiting the flow-through-cell using a graduated container and a stop watch, and record the measurement. Measure and record the water level. Check flow-through-cell for gas bubbles and sediment; if present, remove them.

10. Repeat Step 9 every 5 minutes or as appropriate until monitoring parameters stabilized. Note at least one flow-through-cell volume must be exchanged between readings. If not, the time interval between readings will need to be increased. Stabilization is achieved when three consecutive measurements are within the following limits:

Turbidity (10% for values greater than 5 NTUs; if three Turbidity values are less than 5 NTUs, consider the values as stabilized),
Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),
Specific Conductance (3%),
Temperature (3%),
pH (± 0.1 unit),
Oxidation/Reduction Potential (±10 millivolts).

If these stabilization requirements do not stabilize in a reasonable time, the probes may have been coated from the materials in the groundwater, from a buildup of sediment in the flow-through-cell, or a gas bubble is lodged in the probe. The cell and the probes will need to be cleaned. Turn-off the probes (not the pump), disconnect the cell from the "T" connector and continue to purge the well. Disassemble the cell, remove the sediment, and clean the probes according to the manufacturer's instructions. Reassemble the cell and connect the cell to the "T" connector. Remove all gas bubbles from the cell, turn-on the probes, and continue the measurements. Record that the time the cell was cleaned.

11. When it is time to collect the groundwater samples, turn-off the monitoring probes, and disconnect the pump's tubing from the "T" connector. If you are using a centrifugal or peristaltic pump check the pump's tubing to determine if the tubing is completely filled with water (no air space).

All samples must be collected and preserved according to the analytical method. VOCs and dissolved gases samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

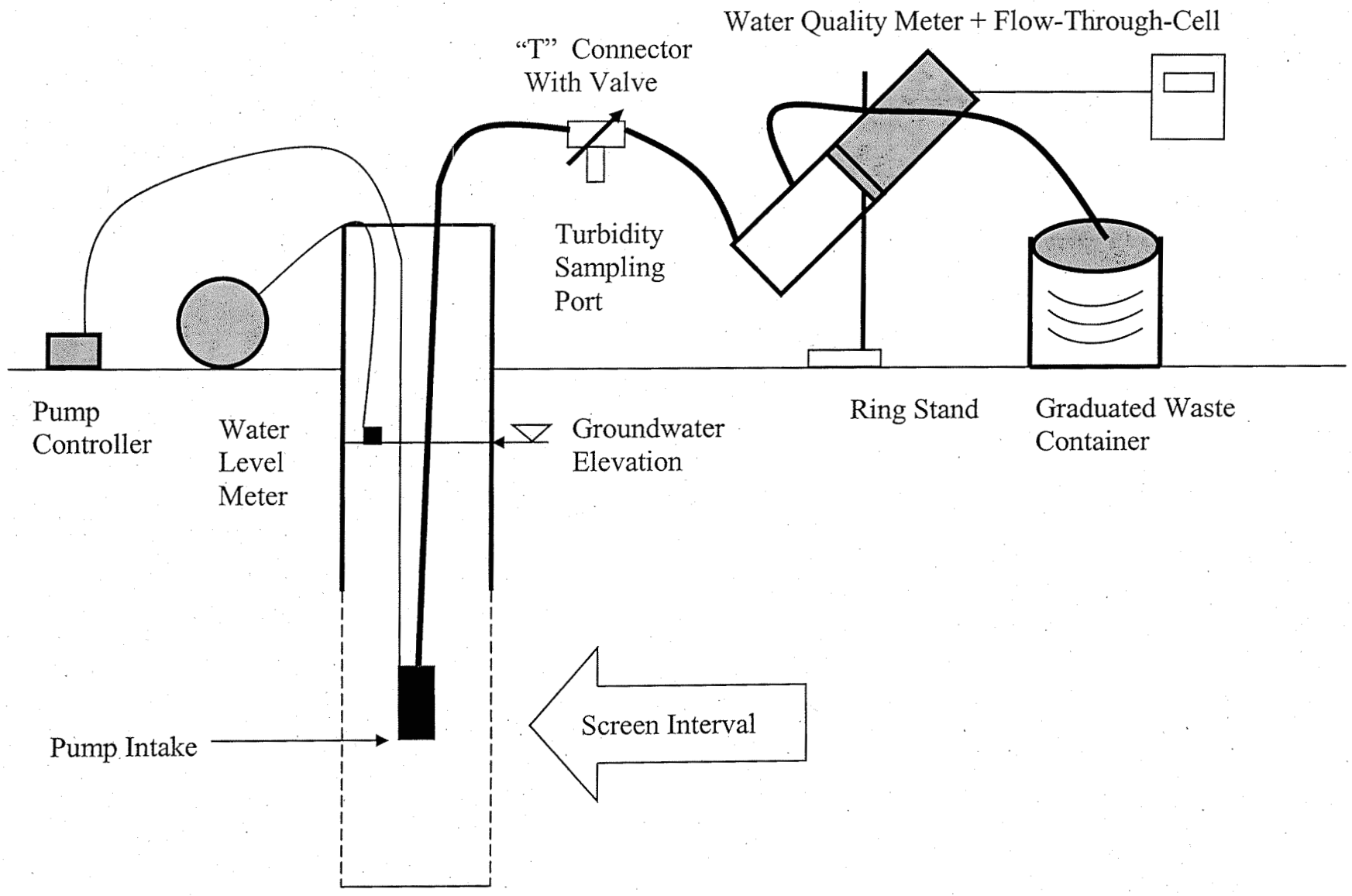
If the pump's tubing is not completely filled with water and the samples are being collected for VOCs and/or dissolved gases analyses using a centrifugal or peristaltic pump, do the following:

> All samples must be collected and preserved according to the analytical method. The VOCs and the dissolved gases (e.g., methane, ethane, ethene, and carbon dioxide) samples are collected last. When it becomes time to collect these samples increase the pump's flow rate until the tubing is completely filled. Collect the samples and record the new flow rate.

12. Store the samples according to the analytical method.

13. Record the total purged volume (graduated waste bucket). Remove the pump from the well and decontaminate the sampling equipment.

Low-Flow Setup Diagram



APPENDIX C
EXAMPLE (Minimum Requirements)
WELL PURGING-FIELD WATER QUALITY MEASUREMENTS FORM

Location (Site/Facility Name)_____
Well Number_____ Date_____
Field Personnel_____
Sampling Organization_____
Identify MP_____

Depth to _____/_____ of screen
(below MP) top bottom
Pump Intake at (ft. below MP)_____
Purging Device; (pump type)_____
Total Volume Purged _____

Clock Time 24 HR	Water Depth below MP ft	Pump Dial[1]	Purge Rate ml/min	Cum. Volume Purged liters	Temp. °C	Spec. Cond.[2] µS/cm	pH	ORP[3] mv	DO mg/L	Turb-idity NTU	Comments
Stabilization Criteria					3%	3%	±0.1	± 10 mv	10%	10%	

1. Pump dial setting (for example: hertz, cycles/min, etc).
2. µSiemens per cm(same as µmhos/cm)at 25 °C.
3. Oxidation reduction potential (ORP)

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number GH-1.2	Page 1 of 9
		Effective Date 09/03	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT		Approved D. Senovich	

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES **2**

5.0 PROCEDURES **2**

5.1 PRELIMINARY EVALUATION 3
5.2 FIELD INSPECTION 3
5.3 WATER LEVEL (HYDRAULIC HEAD) MEASUREMENTS 4
5.3.1 General 4
5.3.2 Water Level Measuring Techniques 5
5.3.3 Methods 5
5.3.4 Water Level Measuring Devices 6
5.3.5 Data Recording 6
5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices 7
5.4 EQUIPMENT DECONTAMINATION 7
5.5 HEALTH AND SAFETY CONSIDERATIONS 7

6.0 RECORDS **7**

ATTACHMENTS

A MONITOIRNG WELL INSPECTON SHEET 8
B GROUNDWATER LEVEL MEASUREMENT SHEET 9

1.0 PURPOSE

The purpose of this procedure is to provide reference information regarding the proper methods for evaluating the physical condition and project utility of existing monitoring wells and determining water levels.

2.0 SCOPE

The procedures described herein are applicable to all existing monitoring wells and, for the most part, are independent of construction materials and methods.

3.0 GLOSSARY

Hydraulic Head - The height to which water will rise in a well.

Water Table - A surface in an unconfined aquifer where groundwater pressure is equal to atmospheric pressure (i.e., the pressure head is zero).

4.0 RESPONSIBILITIES

Site Geologist/Hydrogeologist - Has overall responsibility for the evaluation of existing wells, obtaining water level measurements and developing groundwater contour maps. The site geologist/hydrogeologist (in concurrence with the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number and location of data points which shall be used for constructing a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - Must have a basic familiarity with the equipment and procedures involved in obtaining water levels and must be aware of any project-specific requirements or objectives.

5.0 PROCEDURES

Accurate, valid and useful groundwater monitoring requires that four important conditions be met:

- Proper characterization of site hydrogeology.
- Proper design of the groundwater monitoring program, including adequate numbers of wells installed at appropriate locations and depths.
- Satisfactory methods of groundwater sampling and analysis to meet the project data quality objectives (DQOs).
- The assurance that specific monitoring well samples are representative of water quality conditions in the monitored interval.

To insure that these conditions are met, adequate descriptions of subsurface geology, well construction methods and well testing results must be available. The following steps will help to insure that the required data are available to permit an evaluation of the utility of existing monitoring wells for collecting additional samples.

5.1 Preliminary Evaluation

A necessary first step in evaluating existing monitoring well data is the study and review of the original work plan for monitoring well installation (if available). This helps to familiarize the site geologist/hydrogeologist with site-specific condition, and will promote an understanding of the original purpose of the monitoring wells.

The next step of the evaluation should involve a review of all available information concerning borehole drilling and well construction. This will allow interpretation of groundwater flow conditions and area geology, and will help to establish consistency between hydraulic properties of the well and physical features of the well or formation. The physical features which should be identified and detailed, if available, include:

- The well identification number, permit number and location by referenced coordinates, the distance from prominent site features, or the location of the well on a map.
- The installation dates, drilling methods, well development methods, past sampling dates, and drilling contractors.
- The depth to bedrock -- where rock cores were not taken, auger refusal, drive casing refusal or penetration test results (blow counts for split-barrel sampling) may be used to estimate bedrock interface.
- The soil profile and stratigraphy.
- The borehole depth and diameter.
- The elevation of the top of the protective casing, the top of the well riser, and the ground surface.
- The total depth of the well.
- The type of well materials, screen type, slot size, and length, and the elevation/depths of the screen, interval, and/or monitored interval.
- The elevation/depths of the tops and bottom of the filter pack and well seals and the type and size.

5.2 Field Inspection

During the onsite inspection of existing monitoring wells, features to be noted include:

- The condition of the protective casing, cap and lock.
- The condition of the cement seal surrounding the protective casing.
- The presence of depressions or standing water around the casing.
- The presence of and condition of dedicated sampling equipment.
- The presence of a survey mark on the inner well casing.

If the protective casing, cap and lock have been damaged or the cement collar appears deteriorated, or if there are any depressions around the well casing capable of holding water, surface water may have infiltrated into the well. This may invalidate previous sampling results unless the time when leakage started can be precisely determined.

The routine physical inspection must be followed by a more detailed investigation to identify other potential routes of contamination or sampling equipment malfunction. Any of these occurrences may invalidate

previously-collected water quality data. If the monitoring well is to be used in the future, considerations shown in the steps described above should be rectified to rehabilitate the well.

After disconnecting any wires, cables or electrical sources, remove the lock and open the cap. Check for the presence of organic vapors with a photoionization detector (PID) or flame-ionization detector (FID) to determine the appropriate worker safety level. The following information should be noted:

- Cap function.
- Physical characteristics and composition of the inner casing or riser, including inner diameter and annular space.
- Presence of grout between the riser and outer protective casing and the existence of drain holes in the protective casing.
- Presence of a riser cap, method of attachment to casing, and venting of the riser.
- Presence of dedicated sampling equipment; if possible, remove such equipment and inspect size, materials of construction and condition.

The final step of the field inspection is to confirm previous hydraulic or physical property data and to obtain data not previously available. This includes the determination of static water levels, total well depth and well obstruction. This may be accomplished using a weighted tape measure which can also be used to check for sediment (the weight will advance slowly if sediment is present, and the presence of sediment on the weight upon removal should be noted). If sediment is present and/or the well has not been sampled in 12 or more months, it should be redeveloped before sampling.

Lastly, as a final step, the location, condition and expected water quality of the wells should be reviewed in light of their usefulness for the intended purpose of the investigation.

See Attachment A, Monitoring Well Inspection Sheet.

5.3 Water Level (Hydraulic Head) Measurements

5.3.1 General

Groundwater level measurements can be made in monitoring wells, private or public water wells, piezometers, open boreholes, or test pits (after stabilization). Groundwater measurements should generally not be made in boreholes with drilling rods or auger flights present. If groundwater sampling activities are to occur, groundwater level measurements shall take place prior to well purging or sampling.

All groundwater level measurements shall be made to the nearest 0.01 foot, and recorded in the site geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B), along with the date and time of the reading. The total depth of the well shall be measured and recorded, if not already known. Weather changes that occur over the period of time during which water levels are being taken, such as precipitation and barometric pressure changes, should be noted.

In measuring groundwater levels, there shall be a clearly-established reference point of known elevation, which is normally identified by a mark on the upper edge of the inner well casing. To be useful, the reference point should be tied in with an established USGS benchmark or other properly surveyed elevation datum. An arbitrary datum could be used for an isolated group of wells, if necessary.

Cascading water within a borehole or steel well casings can cause false readings with some types of sounding devices (chalked line, electrical). Oil layers may also cause problems in determining the true water level in a well. Special devices (interface probes) are available for measuring the thickness of oil layers and true depth to groundwater, if required.

Water level readings shall be taken regularly, as required by the site geologist/hydrogeologist. Monitoring wells or open-cased boreholes that are subject to tidal fluctuations should be read in conjunction with a tidal chart (or preferably in conjunction with readings of a tide staff or tide level recorder installed in the adjacent water body); the frequency of such readings shall be established by the site hydrogeologist. All water level measurements at a site used to develop a groundwater contour map shall be made in the shortest practical time to minimize affects due to weather changes.

5.3.2 Water Level Measuring Techniques

There are several methods for determining standing or changing water levels in boreholes and monitoring wells. Certain methods have particular advantages and disadvantages depending upon well conditions. A general description of these methods is presented, along with a listing of various advantages and disadvantages of each technique. An effective technique shall be selected for the particular site conditions by the site geologist/hydrogeologist.

In most instances, preparation of accurate potentiometric surface maps require that static water level measurements be obtained to a precision of 0.01 feet. To obtain such measurements in individual accessible wells, electrical water level indicator methods have been found to be best, and thus should be utilized. Other, less precise methods, such as the popper or bell sound, or bailer line methods, should be avoided. When a large number of (or continuous) readings are required, time-consuming individual readings are not usually feasible. In such cases, it is best to use a pressure transducer.

5.3.3 Methods

Water levels can be measured by several different techniques, but the same steps shall be followed in each case. The proper sequence is as follows:

1. Check operation of recording equipment above ground. Prior to opening the well, don personal protective equipment, as required. Never remove an air-tight lock (such as a J-plug) with your face over the well. Pressure changes within the well may explosively force the cap off once loosened.

2. Record all information specified below in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B):

    - Well number.
    - Water level (to the nearest 0.01 foot). Water levels shall be taken from the surveyed reference mark on the top edge of the inner well casing. If the J-plug was on the well very tightly, it may take several minutes for the water level to stabilize.
    - Time and day of the measurement.
    - Thickness of free product if present.

Water level measuring devices with permanently marked intervals shall be used. The devices shall be free of kinks or folds which will affect the ability of the equipment to hang straight in the well pipe.

5.3.4 Water Level Measuring Devices

Electric Water Level Indicators

These are the most commonly used devices and consist of a spool of small-diameter cable and a weighted probe attached to the end. When the probe comes in contact with the water, an electrical circuit is closed and a meter, light, and/or buzzer attached to the spool will signal the contact.

There are a number of commercial electric sounders available, none of which is entirely reliable under all conditions likely to occur in a contaminated monitoring well. In conditions where there is oil on the water, groundwater with high specific conductance, water cascading into the well, steel well casing, or a turbulent water surface in the well, measuring with an electric sounder may be difficult.

For accurate readings, the probe shall be lowered slowly into the well adjacent to the survey mark on the inner well casing. The electric tape is read (to the nearest 0.01 ft.) at the measuring point and recorded where contact with the water surface was indicated.

Popper or Bell Sounder

A bell- or cup-shaped weight that is hollow on the bottom is attached to a measuring tape and lowered into the well. A "plopping" or "popping" sound is made when the weight strikes the surface of the water. An accurate reading can be determined by lifting and lowering the weight in short strokes, and reading the tape when the weight strikes the water. This method is not sufficiently accurate to obtain water levels to 0.01 feet, and thus is more appropriate for obtaining only approximate water levels quickly.

Pressure Transducer

Pressure transducers can be lowered into a well or borehole to measure the pressure of water and therefore the water elevation above the transducer. The transducer is wired into a recorder at the surface to record changes in water level with time. The recorder digitizes the information and can provide a printout or transfer the information to a computer for evaluation (using a well drawdown/recovery model). The pressure transducer should be initially calibrated with another water level measurement technique to ensure accuracy. This technique is very useful for hydraulic conductivity testing in highly permeable material where repeated, accurate water level measurements are required in a very short period of time. A sensitive transducer element is required to measure water levels to 0.01 foot accuracy.

Borehole Geophysics

Approximate water levels can be determined during geophysical logging of the borehole (although this is not the primary purpose for geophysical logging and such logging is not cost effective if used only for this purpose). Several logging techniques will indicate water level. Commonly-used logs which will indicate saturated/unsaturated conditions include the spontaneous potential (SP) log and the neutron log.

5.3.5 Data Recording

Water level measurements, time, data, and weather conditions shall be recorded in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet. All water level measurements shall be measured from a known reference point. The reference point is generally a marked point on the upper edge of the inner well casing that has been surveyed for an elevation. The exact reference point shall be marked with permanent ink on the casing since the top of the casing may not be entirely level. It is important to note changes in weather conditions because changes in the barometric pressure may affect the water level within the well.

5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices

All groundwater level measurement devices must be cleaned before and after each use to prevent cross contamination of wells. Manufacturer's instructions for cleaning the device shall be strictly followed. Some devices used to measure groundwater levels may need to be calibrated. These devices shall be calibrated to 0.01 foot accuracy and any adjustments/corrections shall be recorded in the field logbook/notebook. After the corrections/adjustments are made to the measuring device and entered in the field logbook/notebook, the corrected readings shall be entered onto the Groundwater Level Measurement Sheet (Attachment B). Elevations will be entered on the sheet when they become available.

5.4 Equipment Decontamination

Equipment used for water level measurements provide a mechanism for potentially cross contaminating wells. Therefore, all portions of a device which project down the well casing must be decontaminated prior to advancing to the next well. Decontamination procedures vary based on the project objectives but must be defined prior to conducting any field activities including the collection of water level data. Consult the project planning documents and SA-7.1 Decontamination of Field Equipment.

5.5 Health and Safety Considerations

Groundwater contaminated by volatile organic compounds may release toxic vapors into the air space inside the well pipe. The release of this air when the well is initially opened is a health/safety hazard which must be considered. Initial monitoring of the well headspace and breathing zone concentrations using a PID or FID shall be performed to determine required levels of protection. Under certain conditions, air-tight well caps may explosively fly off the well when the pressure is relieved. Never stand directly over a well when uncapping it.

6.0 RECORDS

A record of all field procedures, tests and observations must be recorded in the site logbook or designated field notebook. Entries in the log/notebook should include the individuals participating in the field effort, and the date and time. The use of annotated sketches may help to supplement the evaluation.

ATTACHMENT A
MONITORING WELL INSPECTION SHEET

Monitoring Well Inspection Sheet

Project Name: _____ Date: _____

Location: _____ Time: _____

Tidally Influenced: Y / N Personnel: _____

Field Measurements				
Well ID	PID Reading PPM	Depth to Water *	Total Depth *	Flush Mt./ Stick-up

Well Construction Details (Taken from construction logs)		
Total Depth *	Ground Elev.	Top/Btm Screen *

Check List:

Riser Pipe Material:
Riser Notched for Surveyors:
Well ID Tag In-place:
Well security:
Photo taken:

Condition of Well:

Protective Case:
Riser:
Well Pad:
Other:

Presence/Evidence of:

Standing Water Around Well:
Existing Sampling Equipment:
Sediment build-up in Well Btm:

Comments:

** = Measurements are from the top of the inner case to the nearest 0.01'*

ATTACHMENT B
GROUNDWATER LEVEL MESUREMENT SHEET

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____
Location: _____ **Personnel:** _____
Weather Conditions: _____ **Measuring Device:** _____
Tidally Influenced: Yes ___ No ___ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Comments

* All measurements to the nearest 0.01 feet, from surveyed mark (unless noted)

Page ____ of ____

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number SA-6.1	Page 1 of 11
		Effective Date 02/04	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject NON-RADIOLOGICAL SAMPLE HANDLING		Approved D. Senovich	

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES **3**

5.0 PROCEDURES **3**

5.1 SAMPLE CONTAINERS 3
5.2 SAMPLE PRESERVATION 3
5.2.1 Overview 4
5.2.2 Preparation and Addition of Reagents 4
5.3 FIELD FILTRATION 5
5.4 SAMPLE PACKAGING AND SHIPPING 6
5.4.1 Environmental Samples 6

6.0 REFERENCES **7**

ATTACHMENTS

A GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS 8
B ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES 9

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (ɣ261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (ɣ261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration		Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER					
Organics (GC&GC/MS)	VOC Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables (Low SVOCs and pesticide/PCBs)	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticide/PCBs)	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
	Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard	Wide-mouth glass	8 oz.	None	14 days
SOIL					
Organics (GC&GC/MS)	VOC	EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables (Low SVOCs and pesticides/PCBs)	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticides/PCBs)	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium	Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard	Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All	Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All	Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR					
Volatile Organics	Low/Medium	Charcoal tube – 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2 [9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5 [10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11],[12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11],[14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

TETRA TECH	STANDARD OPERATING PROCEDURES	Number SA-1.1	Page 1 of 34
		Effective Date 04/07/2008	Revision 7
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING		Approved Tom Johnston T E Johnston	

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS **3**

5.0 HEALTH AND SAFETY **4**

6.0 PROCEDURES **5**

6.1 General 5
6.2 Sampling, Monitoring, and Evacuation Equipment 7
6.3 Calculations of Well Volume 8
6.4 Evacuation of Static Water – Purging 9
6.4.1 General 9
6.4.2 Evacuation Devices 9
6.5 Onsite Water Quality Testing 12
6.5.1 Measurement of pH 13
6.5.2 Measurement of Specific Conductance 15
6.5.3 Measurement of Temperature 16
6.5.4 Measurement of Dissolved Oxygen 17
6.5.5 Measurement of Oxidation-Reduction Potential 19
6.5.6 Measurement of Salinity 20
6.5.7 Measurement of Turbidity 21
6.6 Sampling 22
6.6.1 Sampling Plan 22
6.6.2 Sampling Methods as Related to Low-Flow Sampling 23
6.7 Low-Flow Purging and Sampling 25
6.7.1 Scope and Application 25
6.7.2 Equipment 25
6.7.3 Purging and Sampling Procedure 26

7.0 REFERENCES **28**

ATTACHMENTS

A PURGING EQUIPMENT SELECTION 29
B GROUNDWATER SAMPLE LOG SHEET 32
C EQUIPMENT CALIBRATION LOG 33
D LOW FLOW PURGE DATA SHEET 34

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the process to be used for purging groundwater monitoring wells prior to sampling, for collecting groundwater samples, and for measuring groundwater quality parameters.

2.0 SCOPE

This document provides information on proper sampling equipment, onsite water quality testing, safety measures to ensure the safety of the field technician(s), and techniques for groundwater sampling. All personnel are encouraged to review the information contained herein to facilitate planning of the field sampling effort. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require modifications to methodology.

3.0 GLOSSARY

Conductivity – Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions and their total concentration, mobility, valence, and relative concentrations and on temperature. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 microSiemens per centimeter (mS/cm) at 14°C.

Dissolved Oxygen (DO) – DO levels in natural and wastewater depend on the physical, chemical, and biochemical activities in the water sample.

Groundwater Sample – A quantity of water removed from the ground, usually via a monitoring well that may or may not be lined with a well casing.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode immersed in water, as referenced against a reference electrode. A reference electrode commonly used in the field is the silver/silver chloride electrode, which has a voltage offset of about 210 mV from the standard hydrogen electrode (SHE). To convert field ORP measurements to equivalent SHE values, approximately 210 mV must be added to the ORP values obtained using the silver/silver chloride electrode. The actual offset depends on the concentration of the potassium chloride (KCl) in the field reference electrode and the temperature. Offsets typically range from 199 (saturated KCl) to 205 (3.5 Molar KCl) to 222 mV (1 Molar KCl) at 25°C and are greater at lower temperatures.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Indicator paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution's pH.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

Salinity – The measurement of dissolved salts in a given mass of solution. Note: most field meters determined salinity automatically from conductivity and temperature. The value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent). The parts per thousand symbol ($^0/_{00}$) is not the same as the percent symbol (%).

Turbidity – Turbidity in water is caused by suspended matter such as clay, silt, and fine organic and inorganic matter. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of groundwater samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager identifies sampling locations.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling, boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Project Hydrogeologist – This individual is responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), equipment to be used, and providing detailed input in this regard to the project planning documents. The project hydrogeologist is also responsible for properly briefing and overseeing the performance of site sampling personnel.

Field Operations Leader (FOL) – This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.
General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Specific safety and health precautions are identified throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas and roadways and along highways.

Methods of avoiding these hazards are provided below.

Knee injuries – Many monitoring wells are installed as flush mounts. Personnel are required to kneel to open these wells and to take groundwater level measurements, etc. This could result in knee injuries from kneeling on stones/foreign objects and general damage due to stress on the joints. To combat this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample wells. To minimize these hazards:

- Pre-survey well locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations – To prevent cuts and lacerations associated with groundwater sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.
- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut -- do not hold them against the opposing hand, a leg, or other body part.

- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.

- DO NOT throw broken glass or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.

- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.

- **Provide a required free space of travel**. Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.

- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.

- Wear high-visibility vests to increase visual recognition by motorists.

- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.

- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

6.1 General

For information derived from a groundwater sample to be useful and accurate, the sample must be representative of the particular zone being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis to keep any changes in water quality parameters to a minimum.

> ***CAUTION***
> A closed well may generate and accumulate gases due to biological degradation, evolution of volatile chemicals from groundwater into the air, or other chemical actions. These gases may also be artificially generated, such as in the case of air sparging or extraction wells, which may take several days to depressurize. See Section 6.6.2 for safety measures to be employed to protect sampling personnel.

Methods for withdrawing samples from completed wells include the use of pumps, compressed air or nitrogen, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water sample due to external influences of the sampling technique(s). In a non-pumping well, there will be little or no vertical mixing of water in the well pipe or casing, and stratification will occur. The well water in the screened section will mix with groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. Concentration gradients resulting from mixing and dispersion processes, layers of variable geologic permeability, and the presence of separate-phase product (e.g., floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase contaminant concentrations in the collected sample compared to what is representative of the integrated water column as it naturally occurs at that point, resulting in the collection of a non-representative sample. To safeguard against collecting non-representative samples, the following approach shall be followed prior to sample acquisition:

CAUTION

Mechanical agitation of well water may cause off-gas generation of volatile contaminants, creating an inhalation exposure to the sampler(s). Where avoiding an inhalation exposure is not possible and mechanical agitation is possible, pump into closed-top containers to control potential air emissions.

1. If possible, position yourself (and the sampling equipment) upwind of the well head.

2. Purge the monitoring well to be sampled prior to obtaining any samples from it. Evacuation of three to five well volumes is recommended prior to sampling, unless low-flow purging and sampling methods are utilized as described in Section 6.7 (Consult the site-specific SAP for exact purging parameters). In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, extensive evacuation prior to sample withdrawal is not as critical as it is in a low-yielding well or in wells containing stagnant water.

3. For wells with low yields that are purged dry during sampling, evacuate the well and allow it to recover to 75 percent of full capacity prior to sample acquisition. If the recovery rate is fairly rapid (generally 300 mL per minute or greater), attempt to continue evacuation until the number of well volumes specified in the SAP is achieved. If this cannot be accomplished, allow recovery to 75 percent of capacity and begin sampling.

CAUTION

For moderate to high-yielding monitoring wells, an evacuation rate that does not cause excessive turbulence in the well should be selected. There is no absolute safeguard against contaminating the sample with stagnant water; hence, special techniques are required for purging to minimize the potential for sample contamination (see below).

4. For moderate to high-yielding monitoring wells, use one of the following purge techniques:

   - Place a submersible pump or the intake line of a surface pump or bailer just below the water surface when removing the stagnant water.

   - While purging and as the water level decreases, lower the pump or intake line as the water level drops in the well. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. After this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.

- Unless otherwise directed, place the intake line of the sampling pump (or the submersible pump itself) near the center of the screened section, and pump approximately one casing volume of water from the well at a low purge rate equal to the well's recovery rate (low-flow sampling).

6.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform to the guidelines in SOP SA-6.1.

The following equipment shall be on hand when sampling groundwater wells (reference SOPs SA-6.1 and SA-7.1):

- Sample packaging and shipping equipment – Coolers for sample shipping and cooling, chemical preservatives, appropriate sampling containers and filler materials, ice, labels, and chain-of-custody documents.
- Field tools and instrumentation
  - Multi-parameter water quality meter with an in-line sample chamber capable of measuring ORP, pH, temperature, DO, specific conductance, turbidity, and salinity, or individual meters (as applicable)
  - pH Paper
  - Camera and film (if appropriate)
  - Appropriate keys (for locked wells)
  - Water level indicator and/or oil-water interface probe if separate-phase product is expected
- Pumps
  - Shallow-well pumps: Centrifugal, bladder, suction, or peristaltic pumps with drop lines and air-lift apparatus (compressor and tubing) where applicable.
  - Deep-well pumps: Submersible pump and electrical power-generating unit, or bladder pumps where applicable.
- Other sampling equipment – Bailers, graduated cylinder, stopwatch, and inert line with tripod-pulley assembly (if necessary).
- Pails – Plastic, graduated.
- Clean paper or cotton towels for cleaning equipment.
- Buckets with lids for collecting purge water.
- Decontamination solutions – Deionized water, potable water, phosphate-free laboratory-grade detergent, and analytical-grade solvent (e.g., pesticide-grade isopropanol), as required.

Ideally, sample withdrawal equipment shall be completely inert, economical, easily cleaned, cleaned prior to use, reusable, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well purging and sample collection.

6.3 Calculations of Well Volume

To ensure that the proper volume of water has been removed from the well prior to sampling, it is first necessary to know the volume of standing water in the well pipe (including well screen where applicable). This volume can be easily calculated by the following method. Calculations shall be entered in the site logbook or field notebook or on a sample log sheet form or equivalent electronic form(s) (see SOP SA-6.3):

1. Obtain all available information on well construction (location, casing, screen, etc.).
2. Determine well or inner casing diameter.
3. Measure and record static water level (depth below ground level or top of casing reference point).
4. Determine depth of well by sounding using a clean, decontaminated, weighted tape measure or water level indicator.
5. Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).
6. Calculate one static well volume in gallons $V = (0.163)(T)(r^2)$

   where: V = Static volume of well in gallons.
   T = Linear feet of water in the well.
   r = Inside radius of well casing in inches.
   0.163 = Conversion factor (compensates for conversion of casing radius from inches to feet and cubic feet to gallons and pi.
7. Per evacuation volumes discussed above, determine the minimum amount to be evacuated before sampling.

Measuring devices may become contaminated when gathering the above information if they are submerged in contaminated water. Decontamination of the tape or water level indicator must be conducted between measurements in different wells as follows:

1. Saturate a paper towel or clean cotton towel with deionized water.
2. As the measuring device is extracted, wipe the tape, changing the cleaning surface frequently.
3. After it is extracted, rinse the probe or tape using a spray bottle of deionized water over a bucket or similar collection container.

Based on the contaminant (oily, etc), it may be necessary to use a soap and water wash and rinse to remove contaminants. Isopropanol can be used on the probe/tape. However, it is recommended that the use of solvents on the tape be minimized because they could degrade the protective covering or possibly remove the scale designations. If isopropanol (or some other solvent) is used, assure that the manufacturer/supplier Material Safety Data Sheet (MSDS) is obtained, kept on site at a readily available location with other MSDSs, and reviewed by personnel prior to the first usage of the solvent. Also, add the substance to the site-specific Hazardous Chemical Inventory list (see Section 5 of the TtNUS Health and Safety Guidance Manual [HSGM], Hazard Communication Program and OSHA Standard 29 CFR 1910.1200).

6.4 Evacuation of Static Water – Purging

6.4.1 General

The amount to be purged from each well will be determined prior to sample collection. This amount will depend on the intent of the monitoring program and the hydrogeologic conditions. Programs to determine overall quality of water resources may require long pumping periods to obtain a sample that is representative of a large volume of the aquifer. The pumped volume may be specified prior to sampling so that the sample can be a composite of a known volume of the aquifer. Alternately, the well can be pumped until parameters such as temperature, specific conductance, pH, and turbidity (as applicable) have stabilized. Onsite measurements of these parameters shall be recorded in the site logbook or field notebook or on standardized data sheets or an equivalent electronic form(s).

6.4.2 Evacuation Devices

The following discussion is limited to those devices commonly used at hazardous waste sites. Attachment A provides guidance on the proper evacuation device to use for given sampling situations. All of these techniques involve equipment that is portable and readily available.

Bailers

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of tubing equipped with a base plate and ball check-valve at the bottom. Bailers are comprised of stainless steel and plastic. They come in a variety of sizes, but the two most often used are 2 inches and 4 inches in diameter. An inert non-absorbent line such as polyethylene rope is used to lower and then raise the bailer to retrieve the sample. As the bailer is lowered into the water column, the ball is pushed up allowing the tube to be filled. When the bailer is pulled upward, the ball seats in the base plate preventing water from escaping.

Advantages of bailers include the following:

- There are few limitations on size and materials used.
- No external power source is needed.
- Bailers are inexpensive and can be dedicated and hung in a well to reduce the chances of cross-contamination.
- Bailers are relatively easy to decontaminate.

Limitations on the use of bailers include the following:

- It is time consuming to remove stagnant water using a bailer.
- Splashing the bailer into the water or transfer of sample may cause aeration.
- The use of a bailer does not permit constant in-line monitoring of groundwater parameters.
- Use of bailers is physically demanding, especially in warm temperatures at personal protection equipment (PPE) levels above Level D.

Safety concerns using a bailer include the following:

- Muscle stress and strain, especially when using 4-inch bailers and when pulling from excessively deep wells.
- Entanglement, possible hand/finger injuries, and rope burns during a sudden release of the bailer back down the well.
- Direct contact with contaminants of concern and sample preservatives when discharging the bailer contents because there is not a high level of control during a direct pour, and splashing and indirect contact with contaminants/preservatives could occur.

Control measures for these hazards are provided in Section 6.6.2.

Suction Pumps

There are many different types of inexpensive suction pumps including centrifugal, diaphragm, and peristaltic pumps. Centrifugal and diaphragm pumps can be used for well evacuation at a fast pumping rate and for sampling at a low pumping rate. The peristaltic pump is a low-volume pump that uses rollers to squeeze flexible tubing to create suction. This tubing can be dedicated to a well to prevent cross-contamination from well to well. Suction pumps are all portable, inexpensive, and readily available. However, because they are based on suction, their use is restricted to areas with water levels within 20 to 25 feet of the ground surface. A significant limitation is that the vacuum created by these pumps can cause loss of dissolved gases and volatile organics. Another limitation of these pumps is that they require a secondary energy source to drive them. Electrically driven pumps may require portable generators as energy sources. Air diaphragm pumps require air compressors and/or compressed gas cylinders to drive them. The advantage of the peristaltic pump is that it will operate from a portable battery source. Safety measures associated with these pumps are provided below.

Air-Lift and Gas-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force groundwater up a sampling tube. These pumps are also relatively inexpensive. Air- or gas-lift samplers are more suitable for well development than for sampling because the samples may be aerated as a result of pump action. Aeration can cause pH changes and subsequent trace metal precipitation or loss of volatile organics.

Submersible Pumps

Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these samplers may be compressed gas or electricity. Operation principles vary, and displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Pumps are available for 2-inch-diameter wells and larger. These pumps can lift water from considerable depths (several hundred feet).

Limitations of this class of pumps include the following:

- They may have low delivery rates.
- Many models are expensive.

- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding of the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time consuming.

Compressed Gases

Safety concerns using compressed gases as an energy source in these pumps are numerous. The nitrogen gas or compressed air is provided in a compressed gas cylinder at a pressure of approximately 2,000 psi. If damaged, these cylinders can become dangerous projectiles. Additionally, a sudden release of a cylinder's contents can involve considerable force that could cause significant damage to the eyes and/or skin. Protective measures include the following:

- Always wear safety impact glasses when handling compressed gases.
- Always administer compressed gases through an appropriate pressure-reducing regulator.
- When clearing the cylinder connection port, open the cylinder valve only enough to clear foreign debris. During this process, always position the cylinder valve so that it faces away from you and others.
- If the cylinder is designed to accept a valve protection cap, always keep that protection cap in place, except the cylinder is connected for use.
- When using the cylinder, lay the cylinder on its side to avoid the potential of it falling and knocking the valve off (and becoming a missile).
- DO NOT use the compressed nitrogen or air to clean clothing or to spray off the skin. Small cuts in the protective layer of the skin may permit the gas to enter into the bloodstream, presenting the potential danger of an embolism.

See the project-specific HASP for additional direction concerning cylinder safe handling procedures pertaining to the safe handling, transportation, and storage of compressed gas cylinders.

Electrical Shock

Even in situations where portable batteries are used, the potential for electrical shock exists. This potential risk is increased in groundwater sampling activities because of the presence of groundwater near the batteries. This potential is also increased in (prohibited) situations where jury-rigging of electrical connections is performed. Other potential hazards occur when field samplers open the hood of a running car to access the battery as a power source. To control these hazards:

- If you are unfamiliar with electrical devices, do not experiment, get help, and get the proper equipment necessary to power your device.
- Use the proper portable power inverters for cigarette lighter connections to minimize the need to access the battery under the hood of your vehicle.
- Use of electrical generators may pose a number of hazards including noise, those associated with fueling, and indirect sample influence.

To minimize or eliminate electrical generator hazards:

- Inspect the generator before use. Ensure that the generator and any extension cords are rated for the intended operation and have a Ground Fault Circuit Interrupter (GFCI) in line to control potential electrical shock.
- Fuel the generator before purging and sampling to avoid loss of power during sampling.
- Fuel engines only when they are turned OFF and have cooled sufficiently to prevent a fire hazard.
- Place the generator and any fuel source at least 50 feet from the well to be sampled to avoid indirect influence to the sample from fuel vapors or emission gases.

Lifting Hazards

This hazard may be experienced when moving containers of purge water, equipment, cylinders, etc. To control these potential hazards:

- Do not fill purge buckets to more than 80 percent of their capacity.
- Obtain a gas cylinder of sufficient size to complete the designated task but not too large to handle. K-size cylinders weigh approximately 135 pounds and are difficult to handle. M-size cylinders weigh approximately 50 pounds and are easier to handle and move.
- When necessary, get help lifting and moving gas cylinders and other heavy objects. Minimize twisting and turning while lifting. If it is necessary to move these cylinders or generators over significant distance, use mechanical means (carts, etc.).
- Use proper lifting techniques as described in Section 4.4 of the HSGM.

6.5 Onsite Water Quality Testing

This section describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific conductance
- Temperature
- DO
- ORP
- Turbidity
- Salinity

This section is applicable for use in an onsite groundwater quality monitoring program to be conducted at a hazardous or nonhazardous waste site. The procedures and equipment described are applicable to groundwater samples and are not, in general, subject to solution interferences from color, turbidity, or colloidal material or other suspended matter.

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Because instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use. Most meters

used to measure field parameters require calibration on a daily basis. Refer to SOP SA-6.3 for an example equipment calibration log.

6.5.1 Measurement of pH

6.5.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken and recorded on the groundwater sample log sheet (Attachment B) or equivalent electronic form.

Two methods are given for pH measurement: the pH meter and pH indicator paper. Indicator paper is used when only an approximation of the pH is required or when pH meter readings need to be verified, and the pH meter is used when a more accurate measurement is needed. The response of a pH meter can be affected by high levels of colloidal or suspended solids, but the effect is generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, or colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

6.5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or alkalinity of the solution created by the addition of the water sample reacting with the indicator compound on the paper. Various types of pH papers are available, including litmus (for general acidity or alkalinity determination) and specific, or narrower range, pH range paper.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to (in this instance, hydrogen) ion activity (which is usually similar to concentration) across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to the ion concentration is generated and measured.

6.5.1.3 Equipment

The following equipment is to be used for obtaining pH measurements:

- A stand-alone portable pH meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Combination electrode with polymer body to fit the above meter. Alternately, a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs.
- Buffer solutions, as specified by the manufacturer. If the buffer solutions are considered hazardous per 29 Code of Federal Regulations (CFR) 1910.1200 (Hazard Communication) or the volumes used are greater than consumer commodity levels, the SSO shall obtain MSDSs from the manufacturer for the specific buffer solutions (see Section 4 of the HSGM regarding the Hazard Communication Program)

- pH indicator paper to cover the pH range 2 through 12.
- Manufacturer's operation manual. All personnel must be familiar with the equipment operation to ensure that the integrity of samples is preserved and that the equipment is operated safely.

6.5.1.4 Measurement Techniques for Field Determination of pH

pH Meter

The following procedure shall be used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

1. Inspect the instrument and batteries prior to initiation of the field effort.
2. Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.
3. If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).
4. Calibrate the meter and electrode(s) on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on a water quality meter calibration log sheet (Attachment C) or equivalent electronic form.
5. Immerse the electrode(s) in the sample. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. The failure of the measurements to stabilize must be clearly noted in the logbook or equivalent electronic form.
6. Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH standard unit. Also record the sample temperature (unless otherwise specified in the SAP, record temperatures to the nearest whole degree Fahrenheit or 0.5 degree Celsius).
7. Rinse the electrode(s) with deionized water.
8. Store the electrode(s) in an accordance with manufacturer's instructions when not in use.

Any visual observation of conditions that may interfere with pH measurement, such as oily materials or turbidity, shall be noted and avoided as much as possible.

pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is determined. To measure the pH with pH paper:

1. Collect a small portion of sample into a clean container.

2. Dip the pH paper into this small portion of sample.

3. Compare the color of the paper to the color chart that is provided with the pH paper and read the corresponding pH from the chart.

4. Record the pH value from the chart on the sampling log sheet.

5. Discard the used pH paper as trash.

6. Discard the small volume of sample that was used for the pH measurement with the other investigative derived waste.

6.5.2 Measurement of Specific Conductance

6.5.2.1 General

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample because temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect specific conductance. Most conductivity meters in use today display specific conductance in units of mS/cm, which is the conductivity normalized to a temperature of 25°C. These are the required units to be recorded on the groundwater sample log field form or equivalent electronic form.

6.5.2.2 Principles of Equipment Operation

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, and the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases, and salts such as hydrochloric acid, sodium carbonate, and sodium chloride, respectively, are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not dissociate in aqueous solution, conduct a current very poorly if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on the ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell, which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

6.5.2.3 Equipment

The following equipment is needed for taking specific conductance measurements:

- Stand-alone portable conductivity meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available that may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirements of the sampling program.

6.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are as follows (calibration shall be conducted according to manufacturer's instructions):

1. Check batteries and calibrate instrument before going into the field.
2. Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form. Potassium chloride solutions with a specific conductance closest to the values expected in the field shall be used for calibration.
3. Rinse the cell with one or more portions of the sample to be tested or with deionized water and shake excess water from the cell.
4. Immerse the electrode in the sample and measure the conductivity.
5. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for troubleshooting assistance.

6.5.3 Measurement of Temperature

6.5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in situ, or as quickly as possible in the field because collected water samples may rapidly equilibrate with the temperature of their surroundings.

6.5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury-filled, dial-type thermometers or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22). In addition, various meters such as specific conductance or DO meters that have temperature measurement capabilities may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

6.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample, use the following procedure:

1. Immerse the thermometer in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples that will undergo subsequent chemical analysis.

2. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

If a temperature meter or probe is used:

1. Calibrate the instrument according to manufacturer's recommendations prior to use.

2. Immerse the meter/probe in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the meter/probe shall not be inserted into samples that will undergo subsequent chemical analysis.

3. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

6.5.4 Measurement of Dissolved Oxygen

6.5.4.1 General

DO levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. In addition, the growth of many aquatic organisms and the rate of corrosivity are dependent on DO concentrations. Thus, analysis for DO is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in situ because concentrations may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of DO meters. Chemical methods of analysis (i.e., Winkler methods) are available but require more equipment and greater sample manipulation. Furthermore, DO meters using a membrane electrode are suitable for highly polluted waters because the probe is completely submersible and is not susceptible to interference caused by color, turbidity, or colloidal material or suspended matter.

6.5.4.2 Principles of Equipment Operation

DO probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between

the two metals, reduction of oxygen to hydroxide ion (OH^-) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode. This rate is proportional to the oxygen concentration in the water being measured.

Because the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, leaving the surface of the solution undisturbed.

DO probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases such as chlorine or with gases such as hydrogen sulfide that are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field logbook and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer. This compensation can counteract some of the temperature effects but not all of them.

6.5.4.3 Equipment

The following equipment is needed to measure DO concentrations:

- A stand-alone portable DO meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

6.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

DO probes differ as to instructions for use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure DO concentrations:

1. Check the DO meter batteries before going to the field.
2. Condition the probe in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.
3. Calibrate the instrument in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
5. Rinse the probe with deionized water.
6. Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane by stirring the sample. Probes without stirrers placed in wells may be moved up and down to achieve the required mixing.

7. Record the DO content and temperature of the sample in a field logbook or on a sample log sheet or equivalent electronic form.

8. Rinse the probe with deionized water.

9. Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable because sample handling is not involved. This however may not always be practical.

Special care shall be taken during sample collection to avoid turbulence that can lead to increased oxygen solubilization and positive test interferences.

6.5.5 Measurement of Oxidation-Reduction Potential

6.5.5.1 General

ORP provides a measure of the tendency of organic or inorganic chemicals to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of reduced to oxidized species in the sample.

6.5.5.2 Principles of Equipment Operation

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as DO, may be correlated with ORP to provide knowledge of the quality of the solution, water, or wastewater.

6.5.5.3 Equipment

The following equipment is needed for measuring the ORP of a solution:

- A combination meter with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

6.5.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring ORP:

1. Check the equipment using the manufacturer's recommended reference solution and check its batteries before going to the field.

2. Thoroughly rinse the electrode with deionized water.

3. If the probe does not respond properly to the recommended reference solution, verify the sensitivity of the electrodes by noting the change in millivolts when the pH of a test solution is altered. The ORP will increase when the pH of a test solution decreases, and the ORP will decrease when the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note that the ORP drops sharply when the caustic is added (i.e., pH increases) thus indicating that the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions or the probe should be replaced.

4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

6.5.6 Measurement of Salinity

6.5.6.1 General

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Most field meters determine salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent).

6.5.6.2 Principles of Equipment Operation

Salinity is determined automatically from the meter's conductivity and temperature readings according to algorithms (such as are found in Standard Methods for the Examination of Water and Wastewater). Depending on the meter, the results are displayed in either ppt or percent. The salinity measurements are carried out in reference to the conductivity of standard seawater (corrected to salinity = 35 ppt).

6.5.6.3 Equipment

The following equipment is needed for salinity measurements:

- A multi-parameter water quality meter capable of measuring conductivity and temperature and converting them to salinity (e.g., Horiba U-22 or YSI 600 series).
- Calibration solution as specified by the manufacturer.
- Manufacturer's operation manual.

6.5.6.4 Measurement Techniques for Salinity

The steps involved in taking salinity measurements are as follows (standardization shall be conducted according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.

2. Check batteries and calibrate the meter before going into the field.

3. Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

4. Rinse the cell with the sample to be tested. This is typically accomplished as the probe is placed in line during the collection of the purge water up to the time of sample acquisition.

5. Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.

6. Rinse the probes with deionized water.

6.5.7 Measurement of Turbidity

6.5.7.1 General

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample. Turbidity in water is caused by suspended matter such as clay, silt, or other finely divided organic and inorganic matter and microscopic organisms including plankton.

It is important to obtain a turbidity reading immediately after taking a sample because irreversible changes in turbidity may occur if the sample is stored too long.

6.5.7.2 Principles of Equipment Operation

Turbidity is measured by the Nephelometric Method, which is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidimeter. Therefore, nephelometric turbidity units (NTUs) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

6.5.7.3 Equipment

The following equipment is needed for turbidity measurements:

- A turbidity meter (e.g., LaMotte 2020) that calibrates easily using test cells with standards of 0.0, 1.0, and 10 NTUs, or a combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).

- Calibration solution and sample tubes, as specified by the manufacturer.

- Manufacturer's operation manual.

6.5.7.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization shall be done according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the instrument before going into the field.
3. Calibrate on a daily basis according to the manufacturer's instructions, and record all pertinent information on a turbidity meter calibration log sheet (Attachment C) or equivalent electronic form.
4. When using the YSI and/or Horiba U-22, rinse the electrode with one or more portions of the sample to be tested or with deionized water.
5. When using the Lamotte 2020, fill the light meter's glass test cell with approximately 5 mL of sample, screw on the cap, wipe off glass to remove all residue that could intercept the instrument's light beam, place the test cell in the light meter, and close the lid.
6. Immerse the electrode in the sample and measure the turbidity.
7. The reading must be taken immediately because suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.
8. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.
9. Rinse the electrode or test cell with deionized water.

6.6 Sampling

6.6.1 Sampling Plan

The sampling approach consisting of the following shall be developed as part of the project planning documents approved prior to beginning work in the field:

- Background and objectives of sampling.
- Brief description of area and waste characterization.
- Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).
- Intended number, sequence, volumes, and types of samples. If the relative degree of contamination between wells is insignificant, a sampling sequence that facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these shall be sampled last to reduce the risk of cross-contamination between wells. In situations where the well is not well-characterized and the nature or extent of airborne contamination is unknown, it is recommended that head space analysis using a photoionization detector (PID) or flame ionization detector (FID) is performed to rate the wells, sampling from least contaminated to most contaminated.

Refer to the project-specific HASP for appropriate information and direction on air monitoring requirements.

- Sample preservation requirements.
- Work schedule.
- List of team members.
- List of observers and contacts.
- Other information, such as the necessity for a warrant or permission of entry, requirements for split samples, access problems, location of keys, etc.
- The FOL shall ensure that the sampling method(s) to be employed is accurately represented in the HASP, indicating the types of sampling to be employed and the hazards. If the methods are not accurately represented, the FOL should rectify this with the HASP author.
- The FOL shall ensure that sampling teams understand the sampling approach that they are to follow. Where sampling teams are made up of personnel from multiple locations, personal sampling experiences may vary. Therefore the FOL shall review project-specific requirements, SOPs, and protocol to be followed. The FOL will conduct periodic surveys to ensure that these methods are being completed per his/her direction.

6.6.2 Sampling Methods as Related to Low-Flow Sampling

The collection of a groundwater sample consists of the following steps:

1. Ensure the safety of the sample location. Take a few minutes to evaluate the area for physical hazards (trip hazards, uneven ground, overhanging branches, etc.) and natural hazards (snakes, bees, spiders, etc.) that may exist in the area or that may have constructed nests in the well head. Snakes often like to sun themselves on concrete well pads. Follow provisions in the project-specific HASP and/or HSGM for addressing natural hazards.
2. As indicated earlier, some monitoring wells have the potential to contain pressurized headspace (e.g., through the generation of gases form contaminated groundwater, due to biological processes, degradation of contaminants, or simply based on location such as near a landfill or in areas that intersect lithological abnormalities) or through intentional artificial means such as those associated with air sparging systems. Injection or extraction wells may be artificially pressurized and may remain so for several days after the system has been turned off. This presents a hazard to people opening these wells. The Field Sampling Technician shall employ the following practices to minimize these hazards:
    - Wear safety glasses to protect the eyes. If site-specific observations and conditions indicate that the wells may be pressurized, wear a full-face shield over the safety impact eye protection.
    - DO NOT place your face or any other part of your body over the well when opening because this may place you in a strike zone.
    - Open the well cover at arms length, then step away and allow the well to off gas and stabilize.

    Follow directions provided in the project-specific HASP, Work Plan and/or Sampling Plan pertaining to the use of volatile chemical detection equipment (PID or FID) within the breathing zone of the sampler

during sampling to determine the need to retreat from the work area and/or for the use of respiratory protection (as specified in the HASP).

3. When proper respiratory protection has been donned, sound the well for total depth and water level (using clean equipment) and record these data on a groundwater sampling log sheet or equivalent electronic form; then calculate the fluid volume in the well pipe (as previously described in this SOP). It is imperative that downhole equipment be adequately decontaminated between wells to prevent cross-contamination. Just as sampling occurs from the least contaminated to the most contaminated, it is also recommended that groundwater level measurements be taken in this manner.

4. Calculate volume of well water to be removed as described in Section 6.3.

5. Select the appropriate purging equipment (see Attachment A to this SOP) or as designated within your Work Plan/Sampling Plan. If an electric submersible pump with packer is chosen, go to Step 10.

6. Lower the purging equipment or intake into the well to a short distance below the water level or mid-screen as indicated in project-specific documentation and begin water removal. Remember that some contaminants are "bottom dwellers," and in these cases, project-specific direction may specify placing the intake just above (1 to 2 feet) the well bottom. Secure the pump intake at the well and secure the effluent at the collection container and begin pumping. The pumping rate will be determined based on the decrease in the water level (see Section 6.7) or as directed in your project-specific documents or this SOP. Purge water is generally collected in a 5-gallon bucket or similar open- or closed-top container. To minimize the potential for spills and back injuries, do not fill 5-gallon buckets beyond approximately 80 percent of their capacity. Dispose of purge water was as indicated in the planning document(s). Where necessary, slow the pumping rate or lower the pump intake as required to maintain submergence.

7. Estimate the approximate rate of discharge frequently and record it on the Low Flow Purge Data Sheet (see Attachment D). Estimate flow rate by noting the amount of discharge in a bucket or graduated cylinder per unit time using a watch with a second hand or a stopwatch.

8. Observe the peristaltic pump tubing intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics.

9. Purge a minimum of three to five casing volumes before sampling (or as directed by the site-specific SAP). In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Allow the well to recover to 75 percent of initial water level before sampling. Do not overfill purge containers because this increases the potential for spills and lifting injuries.

10. If sampling using a submersible pump, lower the pump intake to mid-screen (or the middle of the open section in uncased wells) and collect the sample. If sampling with a bailer, lower the bailer to just below the water surface.

11. For pump and packer assemblies only: Lower the assembly into the well so that the packer is positioned just above the screen or open section. Inflate the packer. Purge a volume equal to at least twice the screened interval (or unscreened open section volume below the packer) before sampling. Packers shall always be tested in a casing section above ground to determine proper inflation pressures for good sealing.

12. If the recovery time of the well is very slow (e.g., 24 hours or greater), sample collection can be delayed until the following day. If the well has been purged early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record this

occurrence in the site logbook or equivalent electronic form. When this occurs, contact the analytical laboratory to alert them that a reduced sample volume(s) will be submitted for analysis.

13. Fill sample containers and preserve and label them as described in SOP SA-6.1. Many sample bottles will contain preservative when they are shipped to the field. In those cases, do not add preservative.

14. Replace the well cap and lock it as appropriate. Make sure the well is readily identifiable as the source of the sample.

15. Process sample containers as described in SOP SA-6.1.

16. Decontaminate equipment as described in SOP SA-7.1.

6.7 Low-Flow Purging and Sampling

6.7.1 Scope and Application

Low-flow purging and sampling techniques may be required for groundwater sampling activities. The purpose of low-flow purging and sampling is to collect groundwater samples that contain "representative" amounts of mobile organic and inorganic constituents in the vicinity of the selected open well interval, at or near natural flow conditions. This minimum-stress procedure emphasizes negligible water level drawdown and low pumping rates to collect samples with minimal alterations in water chemistry. This procedure is designed primarily to be used in wells with a casing diameter of 1 inch or more and a saturated screen length, or open interval, of 10 feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semivolatile organic compounds, pesticides, polychlorinated biphenyls [PCBs], metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This low-flow procedure is not designed for collection of non-aqueous phase liquid samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs).

This procedure is flexible for various well construction types and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than 10 NTUs and to achieve a water level drawdown of less than 0.3 foot during purging and sampling. If these goals cannot be achieved, sample collection can take place provided that the remaining criteria in this procedure are met.

6.7.2 Equipment

The following equipment is required (as applicable) for low-flow purging and sampling:

- Adjustable rate submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon).

- Disposable clear plastic bottom-filling bailers to be used to check for and obtain samples of LNAPLs or DNAPLs.

- Tubing – Teflon, Teflon-lined polyethylene, polyethylene, polyvinyl chloride (PVC), Tygon, or stainless steel tubing can be used to collect samples for analysis, depending on the analyses to be performed and regulatory requirements.

- Water level measuring device with 0.01-foot accuracy (electronic devices are preferred for tracking water level drawdown during all pumping operations).

- Interface probe.
- Flow measurement supplies.
- Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples.
- Indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Use of a flow-through cell is recommended. Optional indicators - ORP, salinity, and DO. A flow-through cell (also referred to as an in-line sample chamber) is required.
- Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s) and other forms (see Attachments B through D) or equivalent electronic form(s).
- Sample bottles.
- Sample preservation supplies (as required by the analytical methods).
- Sample tags and/or labels.
- Well construction data, location map, field data from last sampling event (if available).
- Field Sampling Plan.
- PID or FID instrument for measuring volatile organic compounds (VOCs) per the HASP.

6.7.3 Purging and Sampling Procedure

1. Open the monitoring well as stated earlier and step away. Prepare sampling equipment while allowing 3 to 5 minutes to allow the water level to reach equilibrium. In situations where VOCs are the primary contaminants of concern, air monitoring of the samplers' breathing zone areas may be required by the HASP (typically with a PID or FID).

2 Measure the water level immediately prior to placing the pump in the well and record the water level on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well.

3. Lower the measuring device further into the well to collect the total depth measurement. Again wait 3 to 5 minutes to allow the well to equilibrate to the initial water level prior to placing the pump or pump intake in the well.

4. Record the total well depth on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well

5. Lower the pump or tubing slowly into the well so that the pump intake is located at the center of the saturated screen length of the well. If possible, keep the pump intake at least 2 feet above the bottom of the well to minimize mobilization of sediment that may be present in the bottom of the well. Collection of turbidity-free water samples may be difficult if there is 3 feet or less of standing water in the well.

6. Start with the initial pump rate set at approximately 0.1 liter per minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust the pumping rates as necessary to prevent drawdown from exceeding 0.3 foot during purging. If no drawdown is noted, the pump rate may be increased (to a maximum of 0.4 liter per minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If groundwater is drawn down below the top of the well screen, purging shall cease or the well shall be pumped to dryness and then allowed to recover before purging continues. Well recovery to 75 percent is necessary prior to sampling. Slow-recovering wells should be identified and purged at the beginning of the workday to maximize field work efficiency. If possible, samples should be collected from these wells within the same workday and no later than 24 hours after the end of purging.

7. Measure the water level in the well every 5 to 10 minutes using the water level meter. Record the well water level on the Low Flow Purge Data Form (Attachment D) or equivalent electronic form.
8. Record on the Low Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, ORP, DO, and salinity or as specified by the approved site-specific planning document) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled/deionized water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form or equivalent electronic form.

9. Estimate the flow rate by noting the amount of discharge in a graduated cylinder per unit time using a watch with a second hand. Remeasure the flow rate any time the pump rate is adjusted and periodically during purging. This will determine if a reduction in rate has occurred due to possible battery depletion.

10. During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections and tighten, repair, or replace them as necessary to achieve a tight connection.

11. Wait until stabilization is achieved, or a minimum of two saturated screen volumes have been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits, then begin sampling:

    - pH ±0.2 standard units
    - Specific conductance ±10%
    - Temperature ±10%
    - Turbidity less than 10 NTUs
    - DO ±10%

12. If the above conditions have not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data Form onto the Groundwater Sample Log Form or equivalent electronic form.

NOTE: VOC samples are preferably collected first, directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

13. If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples:

- Collect samples for non-VOC analyses first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample for VOCs, and record the new flow rate.

- Reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel) or clamp, which should reduce the flow rate by constricting the end of the tubing. Proceed with sample collection.

- Insert a narrow-diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing, then collect the sample from the narrow diameter tubing.

- Prepare samples for shipping as per SOP SA-6.1.

7.0 REFERENCES

American Public Health Association, 1989. Standard Methods for the Examination of Water and Wastewater, 17th Edition, APHA, Washington, D.C.

Barcelona, M. J., J. P. Gibb and R. A. Miller, 1983. A Guide to the Selection of Materials for Monitoring Well Construction and Groundwater Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

Johnson Division, UOP, Inc. 1975. Ground Water and Wells, A Reference Book for the Water Well Industry. Johnson Division, UOP, Inc., Saint Paul, Minnesota.

Nielsen, D. M. and G. L. Yeates, 1985. A Comparison of Sampling Mechanisms Available for Small-Diameter Ground Water Monitoring Wells. Ground Water Monitoring Review 5:83-98.

Scalf, M. R., J. F. McNabb, W. J. Dunlap, R. L. Crosby and J. Fryberger, 1981. Manual of Ground Water Sampling Procedures. R. S. Kerr Environmental Research Laboratory, Office of Research and Development, U.S. EPA, Ada, Oklahoma.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020.

U.S. EPA, 1980. Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities. Office of Solid Waste, United States Environmental Protection Agency, Washington, D.C.

U.S. EPA, 1994. Groundwater Sampling Procedure - Low Flow Purge and Sampling (Draft Final). U.S. Environmental Protection Agency, Region I.

U.S. Geological Survey, 1984. National Handbook of Recommended Methods for Water Data Acquisition, Chapter 5: Chemical and Physical Quality of Water and Sediment. U.S. Department of the Interior, Reston, Virginia.

ATTACHMENT A

PURGING EQUIPMENT SELECTION

Diameter Casing		Bailer	Peristaltic Pump	Vacuum Pump	Air-lift	Diaphragm "Trash" Pump	Submersible Diaphragm Pump	Submersible Electric Pump	Submersible Electric Pump w/Packer
1.25-Inch	Water level <25 feet	X	X	X	X	X			
	Water Level >25 feet	X			X				
2-Inch	Water level <25 feet	X	X	X	X	X	X		
	Water Level >25 feet	X			X		X		
4-Inch	Water level <25 feet	X	X	X	X	X	X	X	X
	Water Level >25 feet	X			X		X	X	X
6-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X
8-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X

ATTACHMENT A

PURGING EQUIPMENT SELECTION

PAGE 2

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
BarCad Systems, Inc.	BarCad Sampler	Dedicated; gas drive (positive displacement)	1.5/16	PE, brass, nylon, aluminum oxide	0-150 with std. tubing	1 liter for each 10-15 feet of submergence	$220-350	Requires compressed gas; custom sizes and materials available; acts as piezometer.
Cole-Parmer Inst. Co.	Master Flex 7570 Portable Sampling Pump	Portable; peristaltic (suction)	<1.0/NA	(not submersible) Tygon®, silicone Viton®	0-30	670 mL/min with 7015-20 pump head	$500-600	AC/DC; variable speed control available; other models may have different flow rates.
ECO Pump Corp.	SAMPLifier	Portable; venturi	<1.5 or <2.0/NA	PP, PE, PVC, SS, Teflon®, Tefzel®	0-100	0-500 mL/min depending on lift	$400-700	AC, DC, or gasoline-driven motors available; must be primed.
Geltek Corp.	Bailer 219-4	Portable; grab (positive displacement)	1.66/38	Teflon®	No limit	1,075 mL	$120-135	Other sizes available.
GeoEngineering, Inc.	GEO-MONITOR	Dedicated; gas drive (positive displacement)	1.5/16	PE, PP, PVC, Viton®	Probably 0-150	Approximately 1 liter for each 10 feet of submergence	$185	Acts as piezometer; requires compressed gas.
Industrial and Environmental Analysts, Inc. (IEA)	Aquarius	Portable; bladder (positive displacement)	1.75/43	SS, Teflon®, Viton®	0-250	0-2,800 mL/min	$1,500-3,000	Requires compressed gas; other models available; AC, DC, manual operation possible.
IEA	Syringe Sampler	Portable; grab (positive displacement)	1.75/43	SS, Teflon®	No limit	850 mL sample volume	$1,100	Requires vacuum and/or pressure from hand pump.
Instrument Specialties Co. (ISCO)	Model 2600 Well Sampler	Portable; bladder (positive displacement)	1.75/50	PC, silicone, Teflon®, PP, PE, Detrin®, acetal	0-150	0-7,500 mL/min	$990	Requires compressed gas (40 psi minimum).
Keck Geophysical Instruments, Inc.	SP-81 Submersible Sampling Pump	Portable; helical rotor (positive displacement)	1.75/25	SS, Teflon®, PP, EPDM, Viton®	0-160	0-4,500 mL/min	$3,500	DC operated.
Leonard Mold and Die Works, Inc.	GeoFilter Small Diameter Well Pump (#0500)	Portable; bladder (positive displacement)	1.75/38	SS, Teflon®, PC, Neoprene®	0-400	0-3,500 mL/min	$1,400-1,500	Requires compressed gas (55 psi minimum); pneumatic or AC/DC control module.
Oil Recovery Systems, Inc.	Surface Sampler	Portable; grab (positive displacement)	1.75/12	acrylic, Detrin®	No limit	Approximately 250 mL	$125-160	Other materials and models available; for measuring thickness of "floating" contaminants.
Q.E.D. Environmental Systems, Inc.	Well Wizard® Monitoring System (P-100)	Dedicated; bladder (positive displacement)	1.66/36	PVC	0-230	0-2,000 mL/min	$300-400	Requires compressed gas; piezometric level indicator; other materials available.

ATTACHMENT A

PURGING EQUIPMENT SELECTION

PAGE 3

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
Randolph Austin Co.	Model 500 Vari-Flow Pump	Portable; peristaltic (suction)	<0.5/NA	(Not submersible) Rubber, Tygon®, or Neoprene®	0-30	See comments	$1,200-1,300	Flow rate dependent on motor and tubing selected; AC operated; other models available.
Robert Bennett Co.	Model 180	Portable; piston (positive displacement)	1.8/22	SS, Teflon®, Delrin® PP, Viton®, acrylic, PE	0-500	0-1,800 mL/min	$2,600-2,700	Requires compressed gas; water level indicator and flow meter; custom models available.
Slope Indicator Co. (SINCO)	Model 514124 Pneumatic Water Sampler	Portable; gas drive (positive displacement)	1.9/18	PVC, nylon	0-1,100	250 mL/flushing cycle	$250-350	Requires compressed gas; SS available; piezometer model available; dedicated model available.
Solinst Canada Ltd.	5W Water Sampler	Portable; grab (positive displacement)	1.9/27	PVC, brass, nylon, Neoprene®	0-330	500 mL	$1,300-1,800	Requires compressed gas; custom models available.
TIMCO Mfg. Co., Inc.	Std. Bailer	Portable; grab (positive displacement)	1.66/Custom	PVC, PP	No limit	250 mL/ft of bailer	$20-60	Other sizes, materials, models available; optional bottom-emptying device available; no solvents used.
TIMCO	Air or Gas Lift Sampler	Portable; gas drive (positive displacement)	1.66/30	PVC, Tygon®, Teflon®	0-150	350 mL/flushing cycle	$100-200	Requires compressed gas; other sizes, materials, models available; no solvents used.
Tole Devices Co.	Sampling Pump	Portable; bladder (positive displacement)	1.38/48	SS, silicone, Delrin®, Tygon®	0-125	0-4,000 mL/min	$800-1,000	Compressed gas required; DC control module; custom built.

Construction Material Abbreviations:

PE	Polyethylene
PP	Polypropylene
PVC	Polyvinyl chloride
SS	Stainless steel
PC	Polycarbonate
EPDM	Ethylene-propylene diene (synthetic rubber)

Other Abbreviations:

NA	Not applicable
AC	Alternating current
DC	Direct current

NOTE: Other manufacturers market pumping devices which could be used for groundwater sampling, though not expressly designed for this purpose. The list is not meant to be all-inclusive and listing does not constitute endorsement for use. Information in the table is from sales literature and/or personal communication. No skimmer, scavenger-type, or high-capacity pumps are included.

Source: Barcelona et al., 1983.

ATTACHMENT B
GROUNDWATER SAMPLE LOG SHEET

GROUNDWATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

Page ___ of ___

Project Site Name: _____
Project No.: _____

[ ] Domestic Well Data
[x] Monitoring Well Data
[ ] Other Well Type: _____
[ ] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[X] Low Concentration
[ ] High Concentration

SAMPLING DATA:

Date: / Time: / Method:	Color Visual	pH Standard	S.C. mS/cm	Temp. Degrees C	Turbidity NTU	DO mg/l	ORP mV	Other NA

PURGE DATA:

Date:	Volume	pH	S.C.	Temp. (C)	Turbidity	DO	ORP	Other
Method:								
Monitor Reading (ppm):			SEE LOW FLOW PURGE DATA SHEET					
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:		Signature(s):
MS/MSD	Duplicate ID No.:	

ATTACHMENT C
EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

ATTACHMENT D
LOW FLOW PURGE DATA SHEET

LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: ____________________
PROJECT NUMBER: ____________________

WELL ID: ____________________
DATE: ____________________

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	S. Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Salinity % or ppt	Comments

SIGNATURE(S): ____________________

PAGE___OF___

TETRA TECH	STANDARD OPERATING PROCEDURES	Number SA-1.2	Page 1 of 21
		Effective Date 03/28/2008	Revision 5
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SURFACE WATER AND SEDIMENT SAMPLING		Approved Tom Johnston T E Johnston	

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE 2

2.0 SCOPE 2

3.0 GLOSSARY 2

4.0 RESPONSIBILITIES 2

5.0 HEALTH AND SAFETY 3

6.0 PROCEDURES 6

6.1 Introduction 6
6.1.1 Surface Water Sampling Equipment 6
6.1.2 Surface Water Sampling Techniques 9
6.2 Onsite Water Quality Testing 10
6.3 Sediment Sampling 10
6.3.1 General 10
6.3.2 Sampling Equipment and Techniques for Bottom Materials 11

7.0 REFERENCES 14

ATTACHMENTS

A SURFACE WATER SAMPLE LOG SHEET 16
B SOIL & SEDIMENT SAMPLE LOG SHEET 17
C GUIDANCE ON SAMPLE DESIGN AND SAMPLE COLLECTION 18

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes procedures and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing or for offsite laboratory analysis.

2.0 SCOPE

The information presented in this document is applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

3.0 GLOSSARY

Analyte – Chemical or radiochemical material whose concentration, activity, or mass is measured.

Composite Sample – A sample representing a physical average of grab samples.

Environmental Sample – A quantity of material collected in support of an environmental investigation that does not require special handling or transport considerations as detailed in SOP SA-6.1.

Grab Sample – A portion of material collected to represent material or conditions present at a single unit of space and time.

Hazardous Waste Sample – A sample containing (or suspected to contain) concentrations of contaminants that are high enough to require special handling and/or transport considerations per SOP SA-6.1.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of soil samples. The Project Manager also has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel in accordance with applicable planning documents.

Field Operations Leader - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that

custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface water and sediment samples. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling and boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding boring and sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, , container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Precautions to preserve the health and safety of field personnel implementing this SOP are distributed throughout. The following general hazards may also exist during field activities, and the means of avoiding them must be used to preserve the health and safety of field personnel:

Bridge/Boat Sampling – Potential hazards associated with this activity include:

- Traffic – one of the primary concerns as samplers move across a bridge because free space of travel is not often provided. Control measures should include:
  - When sampling from a bridge, if the samplers do not have at least 6 feet of free travel space or physical barriers separating them and the traffic patterns, the HASP will include a Traffic Control Plan.
  - The use of warning signs and high-visibility vests are required to warn oncoming traffic and to increase the visibility of sample personnel.
- Slips, trips, and falls from elevated surfaces are a primary concern. Fall protection shall be worn when or if samplers must lean over a rail to obtain sample material. A Fall Protection Competent

Person (in accordance with Occupational safety and Health Administration [OSHA] fall protection standards) must be assigned to ensure that fall protection is appropriately and effectively employed

- Water hazards/drowning – if someone enters the water from an elevated surface (such as a bridge or dock) and when sampling from a boat. To minimize this potential, personnel shall wear United states Coast Guard (USCG)-approved floatation devices, and the sampling crew must also have on hand a Type IV Throwable Personal Floatation Device with at least 90 feet of 3/8-inch rope. See Section 5.5.2 of this SOP.

- Within the HASP, provisions will also be provided concerning the requirement of a Safe Vessel Certification or the necessity to conduct a boat inspection prior to use. In addition, the HASP shall also specify requirements as to whether the operator must be certified as a commercial boat operator and whether members of the sampling team must have a state-specific safe boating certification.

Entering Water to Collect Samples – Several hazards are associated with this activity and can be mitigated as follows:

- Personnel must wear a USCG-approved Floatation Device (selected and identified in the HASP). The SSO shall ensure that the device selected is in acceptable condition and suitable for the individual using it. This includes consideration of the weight of the individual.

- Lifelines shall be employed from a point on the shore. This activity will always be conducted with a Buddy. See Section 6.5.2.

- Personnel shall carry a probe to monitor the bottom ahead of them for drop offs or other associated hazards.

- The person in the water shall exercise caution concerning the path traveled so that the lifeline does not become entangled in underwater obstructions such as logs, branches, stumps, etc., thereby restricting its effectiveness in extracting the person from the water.

- Personnel shall not enter waters on foot in situations where natural hazards including alligators, snakes, as well as sharks, gars, and other predators within inland waterways may exist.

- In all cases, working along and/or entering the water during high currents or flood conditions shall be prohibited.

- Personnel shall not enter bodies of water where known debris exists that could result in injuries from cuts and lacerations.

Sampling in marshes or tidal areas in some instances can be accomplished using an all-terrain vehicle (ATV). This is not the primary recommended approach because the vehicle may become disabled, or weather conditions or tidal changes could result in environmental damage as well as loss of the vehicle. The primary approach is recommended to be on foot where minimal disturbance would occur. The same precautions specified above with regard to sediment disturbance apply as well as the previously described safety concerns associated with natural hazards. The natural hazards include alligators, bees (nests in dead falls and tree trunks), snakes, etc. In addition, moving through and over this terrain is difficult and could result in muscle strain and slips, trips, and falls. Common sense dictates that the sampler selects the most open accessible route over moderate terrain. Move slowly and deliberately through challenging terrain to minimize falls. Mud boots or other supportive PPE should be considered and specified in the HASP to permit samplers to move over soft terrain with the least amount of effort. In these situations, it is also recommended, as the terrain allows, that supplies be loaded and transported in a sled over the soft ground.

Working in these areas, also recognize the following hazards and means of protection against them:

Insects are also a primary concern. These include mosquitoes, ticks, spiders, bees, ants, etc. The HASP will identify those particular to your area. Typical preventative measures include:

- Use insect repellant. Approval of various repellants should be approved by the Project Chemist or Project Manager.
- Wearing light-colored clothing to control heat load due to excessive temperatures. In addition, it makes it easier to detect crawling insects on your clothing.
- Taping pants to boots to deny access. Again, this is recommended to control access to the skin by crawling insects. Consultation with the Project Health and Safety Officer SSO/Health and Safety Manager is recommended under extreme heat loads because this will create conditions of heat stress.
- Performing a body check to remove insects. The quicker you remove ticks, the less likely they will become attached and transfer bacteria to your bloodstream. Have your Buddy check areas inaccessible to yourself. This includes areas such as the upper back and between shoulder blades where it is difficult for you to examine and even more difficult for you to remove.

Safety Reminder

If you are allergic to bee or ant stings, it is especially critical that you carry your doctor-recommended antidote with you in these remote sampling locations due to the extended time required to extract incapacitated individuals as well as the effort required to extract them. In these scenarios, instruct your Buddy in the proper administration of the antidote. In all cases, if you have received a sting, administer the antidote regardless of the immediate reaction, evacuate, and seek medical attention as necessary. The FOL and/or SSO will determine when and if you may return to the field based on the extent of the immune response and hazards or potential hazards identified in these locations. To the FOL and SSO, this is a serious decision you have to make as to whether to take someone vulnerable to these hazards into a remote location where you may not be able to carry them out. Consider it wisely.

Poisonous Plants – To minimize the potential of encountering poisonous plants in the field, at least one member of the field team needs to have basic knowledge of what these plants look like so that they can be recognized, pointed out to other field personnel, and avoided if at all possible. If the field team cannot avoid contact and must move through an area where these plants exist, the level of personal protective equipment (PPE) shall include Tyvek coveralls and enhanced decontamination procedures for the removal of oils from the tooling and/or equipment.

Temperature-Related Stress – Excessively cold temperatures may result in cold stress, especially when entering the water either intentionally or by accident. Provisions for combating this hazard should be maintained at the sample location during this activity. Excessively hot temperatures may result in heat stress especially in scenarios where equipment is packed through the marsh.

Because all of these activities are conducted outside, electrical storms are a significant concern. The following measures will be incorporated to minimize this hazard:

- Where possible, utilize commercial warning systems and weather alerts to detect storms moving into the area.

- If on or in the water, get out of the water. Move to vehicles or preferably into enclosed buildings with plumbing and wiring.

- Where warning systems are not available, follow the 30/30 Rule (i*f there are less than 30 seconds between thunder and lightning, go inside for at least 30 minutes after the last thunder).*

See Section 4.0 of the Health and Safety Guidance Manual (HSGM) for additional protective measures.

6.0 PROCEDURES

6.1 Introduction

Collecting a representative sample of surface water or sediment may be difficult because of water movement, stratification, or heterogeneous distribution of the targeted analytes. To collect representative samples, one must standardize sampling methods related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples. Consult Appendix C for guidance on sampling that should be considered during project planning and that may be helpful to field personnel.

6.1.1 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type to be acquired. In general, the most representative samples are obtained from mid-channel at a stream depth of 0.5 foot in a well-mixed stream; however, project-specific planning documents will address site-specific sampling requirements including sample collection points and sampling equipment. The most frequently used samplers include the following:

- Peristaltic pump
- Bailer
- Dip sampler
- Weighted bottle
- Hand pump
- Kemmerer
- Depth-integrating sampler

The dip sampler and weighted bottle sampler are used most often, and detailed discussions for these devices and the Kemmerer sampler are addressed subsequently in this section.

The criteria for selecting a sampler include:

1. Disposability and/or easy decontamination.

2. Inexpensive cost (if the item is to be disposed).

3. Ease of operation.

4. Non-reactive/non-contaminating properties - Teflon-coated, glass, stainless-steel or polyvinyl chloride (PVC) sample chambers are preferred (in that order).

Measurements collected for each sample (grab or each aliquot collected for compositing) shall include but not be limited to:

- Specific conductance
- Temperature
- pH
- Dissolved oxygen

Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field notebook or on sample log sheets (see Attachment A) or an equivalent electronic form(s). These analyses may be selected to provide information on water mixing/stratification and potential contamination. Various types of water bodies have differing potentials for mixing and stratification.

In general, the following equipment if necessary for obtaining surface water samples:

- Required sampling equipment, which may include a remote sampling pole, weighted bottle sampler, Kemmerer sampler, or other device.

- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.

- Required PPE as directed in the project-specific planning document, which may include:

  - Nitrile surgeon's or latex gloves (layered as necessary).

  - Safety glasses.

  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.

> ***Safety Reminder***
> The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).

  - Required decontamination equipment.

  - Required sample containers.

  - Sealable polyethylene bags (e.g., Ziploc® baggies).

  - Heavy-duty cooler.

  - Ice.

- Paper towels and garbage bags.

- Chain-of-custody records and custody seals.

Dip Sampling

Specific procedures for collecting a dip or grab sample of surface water can vary based on site-specific conditions (e.g., conditions near the shore and how closely a sampler can safely get to the shore). The general procedure for collecting a sample using a pole or directly from the water body is as follows:

1. If using a remote sampling pole, securely attach the appropriate sample container to a pole of sufficient length to reach the water to be sampled. Samples for volatile analysis should be collected first. Use PPE as described in the HASP. When sample containers are provided pre-preserved or if the pole cannot accommodate a particular sample container, use a dedicated, clean, unpreserved bottle/container for sampling and transfer to an appropriately preserved container.

2. Remove the cap. Do not place the cap on the ground or elsewhere where it might become contaminated.

3. Carefully dip the container into the water just below the surface (or as directed by project-specific planning documents), and allow the bottle to fill. Sample bottles for volatile analysis must be filled with no headspace. Avoid contacting the bottom of the water body because this will disturb sediment that may interfere with the surface water sample.

4. Retrieve the container and carefully replace the cap securely. If using a container other than the sample bottle, pour the water from that container into the sample bottle and replace the cap securely.

5. Use a clean paper towel to clean and dry the outside of the container.

6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Constituents measured in grab samples collected near the water surface are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration distributed throughout the water column and in the cross section. Therefore, as possible based on site conditions, the sampler may be required to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

CAUTION

In areas prone to natural hazards such as alligators and snakes, etc., always use a buddy as a watch. Always have and use a lifeline or throwable device to extract persons who could potentially fall into the water. Be attentive to the signs, possible mounds indicating nests, and possible slides into the water. Remember that although snakes are typically encountered on the ground, it is not unheard of to see them on low-hanging branches. Be attentive to your surroundings because these may indicate that hazards are nearby.

Weighted Bottle Sampling

A grab sample can also be collected using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of glass or plastic bottle with a stopper, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).

2. When the desired depth is reached, pull out the stopper with a sharp jerk of the stopper line.

3. Allow the bottle to fill completely, as evidenced by the absence of air bubbles.

4. Raise the sampler and cap the bottle.

5. Use a paper towel to clean and dry the outside of the container. This bottle can be used as the sample container as long as the bottle is an approved container type.

6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Kemmerer Sampler

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon-coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while it is lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth.

2. When the desired depth is reached, send down the messenger to close the cylinder and then raise the sampler.

3. Open the sampler valve to fill each sample bottle (filling bottles for volatile analysis first).

4. Use a paper towel to clean and dry the outside of the container.

5. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

6. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

6.1.2 Surface Water Sampling Techniques

Samples collected during site investigations may be grab samples or composite samples. The following general procedures apply to various types of surface water collection techniques:

- If a clean, pre-preserved sample container is not used, rinse the sample container least once with the water to be sampled before the sample is collected. This is not applicable when sample containers are provided pre-preserved because doing so will wash some or all of the preservative out of the bottle.

- For sampling moving water, collect the farthest downstream sample first, and continue sample collection in an upstream direction. In general, work from zones suspected of low contamination to zones of high contamination.

- Take care to avoid excessive agitation of the water because loss of volatile constituents could result.

- When obtaining samples in 40 mL vials with septum-lined lids for volatile organics analysis, fill the container completely (with a meniscus) to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. Turn the vial upside down and tap gently on your wrist to check for air bubbles. If air bubbles rise in the bottle, add additional sample volume to the container.

- Do not sample at the surface, unless sampling specifically for a known constituent that is immiscible and on top of the water. Instead, invert the sample container, lower it to the approximate depth, and hold it at about a 45-degree angle with the mouth of the bottle facing upstream.

6.2 Onsite Water Quality Testing

Onsite water quality testing shall be conducted as described in SOP SA-1.1.

6.3 Sediment Sampling

6.3.1 General

If composite surface water samples are collected, sediment samples are usually collected at the same locations as the associated surface water samples. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body, in a depositional area if possible based on sample location restraints (see below), unless the SAP states otherwise.

Generally, coarser-grained sediments are deposited near the headwaters of reservoirs. Bed sediments near the center of a water body will be composed of fine-grained materials that may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathymetry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled, in general, and areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials shall be generally avoided. Follow instructions in the SAP, as applicable.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with

respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if water column concentrations are less than detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

Samples collected for volatile organic compound (VOC) analysis must be collected prior to any sample homogenization. Regardless of the method used for collection, the aliquot for VOC analysis must be collected directly from the sampling device (hand auger bucket, scoop, trowel), to the extent practical. If a device such as a dredge is used, the aliquot should be collected after the sample is placed in the mixing container prior to mixing.

In some cases, the sediment may be soft and not lend itself to collection by plunging Encore™ or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sampling device, (Encore™ barrel or syringe) prior to sample collection, and carefully place the sediment in the device, filling it fully with the required volume of sample.

On active or former military sites, ordnance items may be encountered in some work areas. Care should be exercised when handling site media (such as if unloading a dredge as these materials may be scooped up). If suspected ordnance items are encountered, stop work immediately, move to shore and notify the Project Manager and Health and Safety Manager.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B or an equivalent electronic form.

6.3.2 Sampling Equipment and Techniques for Bottom Materials

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

SAFETY REMINDER

The following health and safety provisions apply when working on/over/near water:

- At least two people are required to be present at the sampling location in situations where the water depth and/or movement deem it necessary, each wearing a USCG-approved Personal Flotation Devices

- A minimum of three people are required if *any* of the following conditions are anticipated or observed:

  - Work in a waterway that is turbulent *or* swift that could sweep a sampler down stream should he or she fall in accidentally.

  - The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples include large/uneven rocks or boulders, dense mud or sediment that could entrap worker's feet, etc.

  - Waterway is tidal, and conditions such as those listed above could rapidly change.

The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered Personal Flotation Device (Throwable Type IV, life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect sediment samples:

- Scoop sampler
- Dredge samplers
- Coring samplers

Each type of sampler is discussed below.

In general, the following equipment if necessary for obtaining sediment samples:

- Required sampling equipment, which may include a scoop sampler, dredge sampler, coring sampler, or stainless steel or pre-cleaned disposable trowel.
- Stainless bowl or pre-cleaned disposable bowl to homogenize sample.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.
  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc® baggies).
  - Heavy-duty cooler.
  - Ice.
  - Paper towels and garbage bags.
  - Chain-of-custody records and custody seals.

Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if the sampler can safely wade to the required location, the easiest and best way to collect a sediment sample is to use a scoop sampler. Scoop sampling also reduces the potential for cross-contamination. The general scoop sampling procedure is as follows:

1. Reach over or wade into the water body.

2. While facing upstream (into the current), scoop the sampler along the bottom in an upstream direction. Although it is very difficult not to disturb fine-grained materials at the sediment-water interface when using this method, try to keep disturbances to a minimum.

Dredge Samplers

Dredges are generally used to sample sediments that cannot easily be obtained using coring devices (e.g., coarse-grained or partially cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger." Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. The three major types of dredges are Peterson, Eckman and Ponar.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends, thus reducing the "shock wave." The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

The general procedure for using dredge samplers is as follows:

1. Gently lower the dredge to the desired depth.

2. When the desired depth is reached, send the messenger down to cable to close the cylinder and then carefully raise the sampler.

3. Open the sampler to retrieve the sediment.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis *prior to* homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

SAFETY REMINDER

Safety concerns using these dredges include lifting hazards, pinches, and compressions (several pinch points exist within the jaws and levers). In all cases, handle the dredge by the rope to avoid capturing fingers/hands.

Coring Samplers

Coring samplers are used to sample vertical columns of sediment. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand-push tubes to electronic vibrational core tube drivers.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed. The sample is withdrawn intact, permitting the removal of only those layers of interest.

In shallow, wadeable waters, the use of a core liner or tube manufactured of Teflon or plastic is recommended for the collection of sediment samples. Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The general procedure to collecting a sediment sample with a core tube is as follows:

1. Push the tube into the substrate until 4 inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction.

2. Cop the top of the tube to provide suction and reduce the chance of losing the sample.

3. Slowly extract the tube so as not to lose sediment from the bottom of the tube. Cap the bottom of the tube before removing it from the water. This will also help to minimize loss of sample.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis prior to homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or boat using different coring devices such as Ogeechee Sand Pounders, gravity cores, and vibrating coring devices.

All three devices utilize a core barrel with a core liner tube system. The core liners can be removed from the core barrel and replaced with a clean core liner after each sample. Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by turning the core tube to its side and gently pouring the liquid out until fine sediment particles appear in the waste liquid. Post-retrieval processing of samples is the same as above.

7.0 REFERENCES

American Public Health Association, 19.99 Standard Methods for the Examination of Water and Wastewater, 20th Edition, APHA, Washington, D.C.

Feltz, H. R., 1980. Significance of Bottom Material Data in Evaluating Water Quality in Contaminants and Sediments. Ann Arbor, Michigan, Ann Arbor Science Publishers, Inc., V. 1, p. 271-287.

Kittrell, F. W., 1969. A Practical Guide to Water Quality Studies of Streams. U.S. Federal Water Pollution Control Administration, Washington, D.C., 135 p.

U.S. EPA, 1984. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-84-017.

U.S. EPA, 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Water Surveillance Branch, USEPA Surveillance and Analytical Division, Athens, Georgia.

U.S. Geological Survey, 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, USGS, Reston, Virginia.

ATTACHMENT A
SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc. **SURFACE WATER SAMPLE LOG SHEET**

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

	Color	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Date: / Time:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	%	NA
Depth:								
Method:								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date: / Time: / Method: / Monitor Reading (ppm):	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)

COMPOSITE SAMPLE DATA:

Date: / Method: / Monitor Readings (Range in ppm):	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD

Duplicate ID No.:

Signature(s):

APPENDIX C
GUIDANCE ON SAMPLING DESIGN AND SAMPLE COLLECTION

C.1 Defining the Sampling Program

Many factors are considered in developing a sampling program for surface water and/or sediment, including study objectives, accessibility, site topography, physical characteristics of the water body (e.g., flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on vertical and lateral mixing within the body of water. For sediment, dispersion depends on bottom current or flow characteristics, sediment characteristics (e.g., density, size), and geochemical properties (that affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes but must also understand the role of fluvial-sediment transport, deposition, and chemical sorption.

C.1.1 Sampling Program Objectives

The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., contaminated runoff). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the water body, direct waste disposal (solid or liquid) into the water body, and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) that encompasses the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the locations of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc. shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation include: (1) moving the sampling location far enough downstream to allow for adequate mixing, or (2) collecting integrated samples in a cross section. Also, non-homogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

C.1.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes, reservoirs, or larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each contaminant would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of stream flow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Stream flow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining stream flow data by direct or indirect methods. Remember these locations are also where you may encounter natural hazards as these are areas where they hunt. Always exercise extreme caution.

C.1.3 Frequency of Sampling

The sampling frequency and objectives of the sampling event will be defined by the project planning documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of a contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples should be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly and during droughts and floods). Samples of bottom material should generally be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

C.2 Surface Water Sample Collection

C.2.1 Streams, Rivers, Outfalls and Drainage Features

Methods for sampling streams, rivers, outfalls, and drainage features (ditches, culverts) at a single point vary from the simplest of hand-sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).

Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be taken depend on the river's width, depth, and discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of dissolved oxygen (DO), pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project planning documents.

C.2.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained. The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, measurement of DO, pH, temperature, etc. is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample if a sample representative of the water column is required. These vertical composites are often collected along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline that is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer that is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several vertical composites with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality because it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, DO, some cations and anions, and light penetration.

C.2.3 Estuaries

Estuarine areas are, by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Knowledge of the estuary type may be necessary to determine sampling locations. Estuaries are generally categorized into one of the following three types dependent on freshwater inflow and mixing properties:

- Mixed Estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically, this type of estuary is shallow and is found in major freshwater sheet flow areas. Because this type of estuary is well mixed, sampling locations are not critical.

- Salt Wedge Estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally back and forth with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.

- Oceanic Estuary - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small, with the preponderance of the fresh-saline water mixing occurring near or at the shore line.

Sampling in estuarine areas is normally based on the tidal phase, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical DO and temperature profiles.

TETRA TECH	STANDARD OPERATING PROCEDURES	Number SA-1.3	Page 1 of 31
		Effective Date 04/072008	Revision 9
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SOIL SAMPLING		Approved Tom Johnston T E Johnston	

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE 2

2.0 SCOPE 2

3.0 GLOSSARY 2

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS 3

5.0 HEALTH AND SAFETY 4

6.0 PROCEDURES 5

6.1 Overview 6
6.2 Soil Sample Collection 6
6.2.1 Procedure for Preserving and Collecting Soil Samples for Volatile Organic Compound Analysis 6
6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses 9
6.2.3 Procedure for Collecting Undisturbed Soil Samples 10
6.3 Surface Soil Sampling 13
6.4 Near-Surface Soil Sampling 14
6.5 Subsurface Soil Sampling With a Hand Auger 15
6.6 Subsurface Soil Sampling with a Split-Barrel Sampler 17
6.7 Subsurface Soil Sampling Using Direct-Push Technology 18
6.8 Excavation and Sampling of Test Pits and Trenches 18
6.8.1 Applicability 18
6.8.2 Test Pit and Trench Excavation 19
6.8.3 Sampling in Test Pits and Trenches 21
6.8.4 Backfilling of Trenches and Test Pits 25
6.9 Records 25

7.0 REFERENCES 26

ATTACHMENTS

A SOIL & SEDIMENT SAMPLE LOG SHEET 28
B SPLIT-SPOON SAMPLER 29
C TEST PIT LOG 30
D REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING 31

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used to collect surface, near-surface, and subsurface soil samples. Additionally, it describes the methods for sampling of test pits and trenches to determine subsurface soil and rock conditions and for recovery of small-volume or bulk samples from pits.

2.0 SCOPE

This document applies to the collection of surface, near-surface, and subsurface soil samples exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites for laboratory testing, onsite visual examination, and onsite testing.

3.0 GLOSSARY

Composite Sample - A composite sample is a combination of more than one grab sample from various locations and/or depths and times that is homogenized and treated as one sample. This type of sample is usually collected when determination of an average waste concentration for a specific area is required. Composite samples shall not be collected for volatile organics analysis.

Confined Space - As stipulated in 29 Code of Federal Regulations (CFR) 1910.146, a confined space means a space that: (1) is large enough and so configured that an employee can bodily enter and perform assigned work; (2) has limited or restricted means for entry or exit (e.g., tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations); and (3) is not designed for continuous employee occupancy. TtNUS considers all confined space as permit-required confined spaces.

Grab Sample - One sample collected at one location and at one specific time.

Hand Auger - A sampling device used to extract soil from the ground.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

Sample for Non-Volatile Analyses - Includes all chemical parameters other than volatile organics (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into resistant materials using a drive weight mounted in the drilling string. A standard split-barrel sampler is typically available in two common lengths, providing either 20-inch or 26-inch longitudinal clearance for obtaining 18-inch or 24-inch-long samples, respectively. These split-barrel samplers commonly range in size from 2 to 3.5 inches OD. The larger sizes are commonly used when a larger volume of sample material is required (see Attachment B).

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine shallow subsurface conditions for engineering, geological, and soil chemistry exploration and/or sampling purposes. These pits are excavated manually or by machine (e.g., backhoe, clamshell, trencher, excavator, or bulldozer).

Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, selecting proposed sampling locations, and selecting field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring, and excavation activities and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation, and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface, near-surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits, and trenches and for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and/or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions that are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Health and safety precautions are identified for individual sample collection procedures throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard or uneven surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas, along roadways and highways.

Methods of avoiding these hazards are provided below.

Knee injuries – If kneeling is required during soil sampling, this could result in knee injuries from stones/foreign objects and general damage due to stress on the joints. To minimize this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.
- Stretch ligaments, tendons and muscles before, during and after. Take breaks as frequently as necessary.
- Report pre-existing conditions to the SSO if you feel this activity will aggravate an existing condition.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample locations. To minimize these hazards:

- Pre-survey sampling locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations - To prevent cuts and lacerations associated with soil sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.

- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut – do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken sample jars or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel**. Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

The following procedures address surface and subsurface sampling.

CAUTION

Each situation must be evaluated individually to determine the applicability and necessity for obtaining a utility clearance ticket/dig permit. Common sense dictates, prior to digging or boring with power equipment, no matter what the depth, or digging by hand in a manner that could damage unprotected underground utilities, that a dig permit is required. See SOP HS-1.0, Utility Locating and Excavation Clearance, for additional clarification. If you do not know or are unsure as to whether a ticket is necessary – **Get the Ticket**.

6.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they migrate to the water table, and can establish the amount of contamination absorbed or adsorbed on aquifer solids that have the potential of contributing to groundwater contamination.

Soil types can vary considerably on a hazardous waste site. These variations, along with vegetation, can affect the rate of contaminant migration through the soil. It is important, therefore, that a detailed record be maintained during sampling operations, particularly noting sampling locations, depths, and such characteristics as grain size, color, and odor. Subsurface conditions are often stable on a daily basis and may demonstrate only slight seasonal variation especially with respect to temperature, available oxygen and light penetration. Changes in any of these conditions can radically alter the rate of chemical reactions or the associated microbiological community, thus further altering specific site conditions. Certain vegetation species can create degradation products that can alter contaminant concentrations in soil. This is why vegetation types and extent of degradation of this foliage must be recorded. To prevent degradation, samples must be kept at their at-depth temperature or lower, protected from direct light, sealed tightly in approved glass containers, and be analyzed as soon as possible after collection. In addition, to the extent possible, vegetation should be removed from the sample.

The physical properties of the soil, its grain size, cohesiveness, associated moisture, and such factors as depth to bedrock and water table, will limit the depth from which samples can be collected and the method required to collect them. It is the intent of this document to present the most commonly employed soil sampling methods used at hazardous waste sites.

6.2 Soil Sample Collection

6.2.1 Procedure for Preserving and Collecting Soil Samples for Volatile Organic Compound Analysis

Samples collected using traditional methods such as collection in a jar with no preservation have been known to yield non-representative samples due to loss of volatile organic compounds (VOCs). To prevent such losses, preservation of samples with methanol or sodium bisulfate may be used to minimize volatilization and biodegradation. This preservation may be performed either in the field or laboratory, depending on the sampling methodology employed. Because of the large number of sampling methods and associated equipment required, careful coordination between field and laboratory personnel is needed.

Soil samples to be preserved by the laboratory are currently being collected using Method SW-846, 5035. For samples preserved in the field, laboratories are currently performing low-level analyses (sodium bisulfate preservation) and high- to medium-level analyses (methanol preservation) depending on the needs of the end user.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

6.2.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for volatile organic analysis that are to be preserved at the laboratory shall be obtained using a hermetically sealed sample vial such as an EnCore™ sampler. Each sample shall be

obtained using a reusable sampling handle (T-handle) that can be provided with the EnCore™ sampler when requested and purchased. Collect the sample in the following manner for each EnCore™ sampler:

1. Scene Safety - Evaluate the area where sampling will occur. Ensure that the area is safe from physical, chemical, and natural hazards. Clear or barricade those hazards that have been identified.

2. Wear the appropriate personal protective equipment (PPE). This will include, at a minimum, safety glasses and nitrile surgeon's gloves. If you must kneel on the ground or place equipment on the surface being sampled, cover the ground surface with plastic to minimize surface contamination of your equipment and clothing. Wear knee pads to protect your knees from kneeling on hard or uneven surfaces.

3. Load the Encore™ sampler into the T-handle with the plunger fully depressed.

4. Expose the area to be sampled using a hand trowel or similar device to remove surface debris.

5. Press the T-handle against the freshly exposed soil surface, forcing soil into the sampler. The plunger will be forced upward as the cavity fills with soil.

6. When the sampler is full, rotate the plunger and lock it into place. If the plunger does not lock, the sampler is not full. This method ensures there is no headspace. Soft soil may require several plunges or forcing soil against a hard surface such as a sample trowel to ensure that headspace is eliminated.

7. Use a paper towel to remove soil from the side of the sampler so a tight seal can be made between the sample cap and the rubber O-ring.

8. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler.

9. Remove any surface soil from the outside of the sampler and place in the foil bag provided with the sampler. Good work hygiene practices and diligent decontamination procedures prevents the spread of contamination even on the outside of the containers.

10. Label the bag with appropriate information in accordance with SOP SA-6.3.

11. Place the full sampler inside a lined cooler with ice and cool to 4˚C ± 2 ˚C. Make sure any required trip blanks and temperature blanks are also in the cooler. Secure custody of the cooler in accordance with SOP SA-6.3.

12. Typically, collect three Encore™ samplers at each location. Consult the SAP or laboratory to determine the required number of Encore™ samplers to be collected.

13. The T-handle shall be decontaminated before moving to the next interval or location using a soap and water wash and rinse, and where applicable, the selected solvent as defined in the project planning documents.

Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler.

After the Encore™samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

6.2.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analyses using both the low-level (sodium bisulfate preservation) and high- to medium-level (methanol preservation) methods.

Safety Reminder

When using chemicals in the field to preserve samples, the FOL and/or SSO must ensure that Materials Safety Data Sheets (MSDSs) have been provided with the chemicals to be used. They also must ensure that these chemicals have been added to the Chemical Inventory List contained within Section 5.0, Hazard Communication, of your Health and Safety Guidance Manual (HSGM). Lastly, but most importantly, the FOL and/or SSO must review the hazards with personnel using these chemicals and ensure that provisions are available for recommended PPE and emergency measures (e.g., eyewash, etc.).

Methanol Preservation (High to Medium Level):

Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40 to 60 mL glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

CAUTION

NEVER attempt to pipette by mouth

In situations where personnel are required to spike the septum using a hypodermic needle, the following provisions for handling sharps must be in place:

- Training of personnel regarding methods for handling of sharps
- Hard-sided containers for the disposal of sharps
- Provisions for treatment in cases where persons have received a puncture wound

Soil shall be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable tube/plunger-type syringe with the tip cut off. The outside diameter of the coring device must be smaller than the inside diameter of the sample bottle neck.

A small electronic balance or manual scale will be necessary for measuring the volume of soil to be added to the methanol-preserved sample bottle. Calibration of the scale shall be performed prior to use and intermittently throughout the day according to the manufacturer's requirements.

The sample should be collected as follows:

1. Weigh the unused syringe and plunger to the nearest 0.01 gram.

2. Pull the plunger back and insert the syringe into the soil to be sampled.

3. Collect 8 to 12 grams of soil by pushing the syringe barrel into the soil.

4. Weigh the sample and adjust until obtaining the required amount of sample.

5. Record the sample weight to the nearest 0.01 gram in the field logbook and/or on the sample log sheet.

6. Extrude the weighed soil sample into the methanol-preserved sample bottle taking care not to contact the sample container with the syringe.

7. If dirty, wipe soil particles from the threads of the bottle and cap. Cap the bottle tightly.

8. After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol.

9. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

Sodium Bisulfate Preservation (Low Level):

> ***CAUTION***
> Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soil containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode. To avoid this hazard or hazards of this type, a small sample aliquot should be subjected to the sample preservative. If it effervesces in an open air environment, utilize an alternative method such as Encore™ or 2-ounce jar.

Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be preserved in the field using the sodium bisulfate method are to be prepared and collected as follows:

1. Add 1 gram of sodium bisulfate to 5 mL of laboratory-grade deionized water in a 40 to 60 mL glass vial with septum-lined lid.

2. Collect the soil sample and record the sample weight to the nearest 0.01 gram in the field logbook or on the sample log sheet as described for methanol preservation

3. Add the weighed sample to the sample vial.

4. Collect duplicate samples using the methanol preservation method on a one-for-one sample basis because it is necessary for the laboratory to perform both low-level and medium-level analyses.

5. Place the samples on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

> ***NOTE***
> If lower detection limits are necessary, an option to field preserving with sodium bisulfate may be to collect EnCore™ samplers at a given sample location. Consult the planning documents to determine whether this is required. If it is, collect samples in accordance with the Encore™ sampling procedure above and then send all samplers to the laboratory to perform the required preservation and analyses.

6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses

Samples collected for non-volatile analyses may be collected as either grab or composite samples as follows:

1. With a stainless steel trowel or other approved tool, transfer a portion of soil to be sampled to a stainless steel bowl or disposable inert plastic tray.

2. Remove roots, vegetation, sticks, and stones larger than the size of a green pea.

3. Thoroughly mix the soil in the bowl or tray to obtain as uniform a texture and color as practicable. The soil type, moisture content, amount of vegetation, and other factors may affect the amount of time required to obtain a properly mixed sample. In some cases, it may be impossible to obtain a uniform sample appearance. Use the field logbook to describe any significant difficulties encountered in obtaining a uniform mixture.

4. Transfer the mixed soil to the appropriate sample containers and close the containers.

5. Label the sample containers in accordance with SOP SA-6.3.

6. Place the containers in a cooler of ice as soon after collection as possible.

7. Prepare the sample shipment and ship the samples in accordance with SOP SA-6.1.

NOTE
Cooling may not be required for some samples depending on the scheduled analyses. Consult the planning documents if in doubt regarding correct sample preservation conditions. When in doubt – Cool to 4˚C.

NOTE
Head space is permitted in soil sample containers for non-volatile analyses to allow for sample expansion.

6.2.3 Procedure for Collecting Undisturbed Soil Samples

NOTE
Use of thin-walled undisturbed tube samplers is restricted by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soil with a consistency in excess of very stiff cannot be penetrated by the sampler. Devices such as Dennison or Pitcher core samplers can be used to obtain undisturbed samples of stiff soil. Using these devices normally increases sampling costs, and therefore their use should be weighed against the need for acquiring an undisturbed sample. These devices are not discussed in this SOP because they are not commonly used.

When it is necessary to acquire undisturbed samples of soil for purposes of engineering parameter analysis (e.g., permeability), a thin-walled, seamless tube sampler (Shelby tube) shall be employed using the following collection procedure:

1. In preparation for sampling utilizing a drill rig, field personnel must complete the following activities:

   - Ensure that all subsurface drilling activities are preceded by a utility clearance for the area to be investigated. This includes activities described in SOP HS-1.0, Utility Location and Excavation Clearance, as well as any location-specific procedures that may apply.

REMEMBER

If you are digging near a marked utility (within the diameter of an underground utility that has been marked plus 18 inches), you must first locate the utility through vacuum extraction or hand digging to ensure that your activities will not damage the utility.

- Complete an Equipment Inspection Checklist for the drill rig or direct-push technology (DPT) rig. This checklist will be provided in the HASP.
- Review the Safe Work Permit prior to conducting the activity.
- Review the activity to be conducted.

2. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and/or clean out the borehole to the desired sampling depth. Be careful to minimize potential disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

CAUTION

The use of bottom-discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Only the use of side-discharge bits is permitted.

3. Determine whether a stationary piston-type sampler is required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used.

4. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal. In addition, the check valve maintains a positive suction within the tube to help retain the sample.

5. A stainless steel tube sampler is typically used to minimize chemical reaction between the sample and the sampling tube.

6. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil with a continuous and rapid motion, without impacting or twisting. If the soil is too hard to penetrate by pushing alone, careful hammering may be used by minimizing drop distance (tapping) of the hammer. Before pulling the tube, turn it at least one revolution to shear the sample off at the bottom. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.

7. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated.

8. Remove disturbed material in the upper end of the tube and measure the length of sample again.

9. After removing at least 1 inch of soil from the lower end, place enough packing material (clean inert material such as paper or cloth) tightly in each end of the Shelby tube and then pour melted wax into each end to make at least a ½-inch wax plug and then add more packing material to fill the voids at both ends.

10. Place plastic caps on the ends, tape the caps in place, and dip the ends in wax to prevent loss of soil.

11. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label.

12. Mark the "up" direction on the side and upper end of the tube with indelible ink.

13. Complete a chain-of-custody form (see SOP SA-6.3) and other required forms (including Attachment A of this SOP).

14. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

CAUTION

To preserve sample integrity do not allow tubes to freeze, and store the samples vertically with the same orientation they had in the ground, (i.e., top of sample is up) in a cool place out of the sun at all times.

CAUTION

A primary concern in the preparation of the wax plugs is the potential for the heat source and melted wax to cause a fire and/or burns. Follow the directions below to prevent injury or fire.

Electrical Heating

Using hot plates to melt the wax is acceptable. In an outdoor setting, make sure a Ground Fault Circuit Interrupter (GFCI) is employed within the electrical circuit. If a portable generator is used, ensure that the generator is an adequate distance from the sampling operation (at least 50 feet). Ensure that the extension cord is rated for the intended load and for outdoor use and is free from recognizable damage. Ensure flammable preservatives are not employed or stored near the hot plate. Although a Hot Work Permit is not required, scene safety evaluation by site personnel of the above elements is. As always, if a fire potential exists, the provisions for extinguishing must be immediately accessible as well as any provisions for first aid measures.

Open Flame

If an open flame is used, the following provisions are necessary:

- Complete a Hot Work Permit and any local permit required for elevated temperature applications. The Hot Work Permit, provided in your HASP, will aid the FOL and/or the SSO in ensuring that fire protection provisions (extinguishers, fire watches, etc.) are in place as well as ensuring that local requirements have been addressed.

- Ensure that water is available to address any wax splashes or contact. If possible, immerse the contacted area. Where this is not possible, run water over the area and apply cold compresses. The need for medical attention or first aid shall be determined on site under the direction of the SSO.

6.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples for subsequent analysis is by use of a stainless steel shovel, hand auger, soil corer, or stainless steel or disposable plastic trowel.

NOTE

Multiple depth intervals are used to describe surface soil. Sometimes surface soil is defined as soil from 0 to 2 inches below ground surface (bgs), and sometimes it is defined as soil from other depths such as 0 to 2 feet bgs. Ensure that the definition of surface soil depth is clear before collecting surface soil samples.

For the purposes of instruction, the terms "surface soil" and "near-surface soil" are used in this SOP as follows:

- Surface soil - 0 to 6 inches bgs
- Near-surface soil - 6 to 18 inches bgs

If these intervals are defined differently in the planning documents, substitute the appropriate depth ranges.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Stainless steel hand auger, soil corer, or shovel.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in project planning document.
- Required PPE.
  - Nitrile surgeon's or latex gloves may be used, layered as necessary.
  - Safety glasses
  - Other – Items identified on the Safe Work Permit may be required based on location-specific requirements such as hearing protection, steel-toed work boots, and a hard hat when working near a drill rig. These provisions will be listed in the HASP or directed by the FOL and/or SSO.

Safety Reminder

The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP)
- Required decontamination equipment
- Required sample container(s)
- Wooden stakes or pin flags

- Sealable polyethylene bags (e.g., Ziploc® baggies)
- Heavy duty cooler
- Ice
- Chain-of-custody records and custody seals

When acquiring surface soil samples, use the following procedure:

1. Place padding or use knee pads when kneeling near the sample location. If necessary, place plastic sheeting to provide a clean surface for sample equipment to avoid possible cross- contamination.

2. Carefully remove vegetation, roots, twigs, litter, etc. to expose an adequate soil surface area to accommodate sample volume requirements.

3. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting surface soil samples for volatile analysis. Surface soil samples for volatile organic analysis should be collected deeper than 6 inches bgs because shallower material has usually lost most of the volatiles through evaporation. Ensure that the appropriate surface soil depth is being analyzed in accordance with the planning document.

4. Using decontaminated sampling tools, thoroughly mix in place a sufficient amount of soil to fill the remaining sample containers. See Section 6.5 of this procedure for hand auger instruction, as needed.

5. Transfer the sample into those containers utilizing a stainless steel trowel.

6. Cap and securely tighten all sample containers.

7. Affix a sample label to each container. Be sure to fill out each label carefully and clearly, addressing all the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

9. Site restoration – Whenever removing sample materials, always restore the surface. It is our intent to leave the area better than we found it. Do NOT create trip hazards in areas when pedestrian traffic may exist.

6.4 Near-Surface Soil Sampling

Collection of samples from near the surface (depth of 6 to 18 inches) can be accomplished with tools such as shovels, hand auger, soil corers, and stainless steel or pre-cleaned disposable trowels and the equipment listed under Section 6.5 of this procedure.

To obtain near-surface soil samples, the following protocol shall be used:

1. With a clean shovel, make a series of vertical cuts in the soil to the depth required to form a square approximately 1 foot by 1 foot.

2. Lever out the formed plug and scrape the bottom of the freshly dug hole with a decontaminated stainless steel or pre-cleaned disposable trowel to remove any loose soil.

3. Follow steps 1 through 9 of Section 6.3.

6.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of stainless steel bucket bits (approximately 6.5 inches long and 2, 2.75, 3.25, and 4 inches in diameter), series of extension rods (available in 2-, 3-, 4- and 5-inch lengths), and a T-handle connected to extension rods and to the auger bucket. A larger-diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then it is withdrawn. The larger-diameter bit is then replaced with a smaller-diameter bit, lowered down the hole, and slowly turned into the soil to the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil either from the surface, or to depths in excess of 12 feet. However, the presence of subsurface rocks and landfill material and collapse of the borehole normally limit sampling depth.

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes)
- Stainless steel mixing bowls
- The equipment listed in Section 6.3
- Miscellaneous hand tools as required to assemble and disassemble the hand auger units

CAUTION

Potential hazards associated with hand augering include:

- Muscle strain and sprain due to over twisting and/or over compromising yourself.
- Equipment failure due to excessive stress on the T-handle or rods through twisting. Failure of any of these components will result in a sudden release and potential injury due to that failure.

As in all situations, any intrusive activities that could damage underground utilities shall be proceeded by a Dig/Excavation permit/ticket. Call the Utility Locating service in the area or your Project Health and Safety Officer for more information. When in doubt – **Get the Ticket!**

To obtain soil samples using a hand auger, use the following procedure:

1. Wearing designated PPE, attach a properly decontaminated bucket bit to a clean extension rod and attach the T-handle to the extension rod.
2. Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.).
3. Twist the bucket into the ground while pushing vertically downward on the auger. The cutting shoes fill the bucket as it is advanced into the ground.
4. As the auger bucket fills with soil, periodically remove any unneeded soil.

5. Add rod extensions as necessary to extend the reach of the auger. Also, note (in a field notebook, boring log, and/or on a standardized data sheet) any changes in the color, texture or odor of the soil as a function of depth. The project-specific planning document (SAP, HASP, etc.) describe requirements for scanning the soil with a real-time air monitoring instrument (e.g., PID, FID, etc.) and recording the measurements.

6. After reaching the desired depth (e.g., the top of the interval to be sampled), slowly and carefully withdraw the apparatus from the borehole to prevent or minimize movement of soil from shallower intervals to the bottom of the hole.

7. Remove the soiled bucket bit from the rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is to be smaller in diameter than the bucket bit employed to initiate the borehole.

8. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.

9. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.

10. Discard the top of the core (approximately 1 inch), which represents any loose material collected by the bucket bit before penetrating the sample material.

11. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting a soil sample for volatile compound analysis directly from the bucket bit.
12. Utilizing a properly decontaminated stainless steel trowel or dedicated disposable trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl.

13. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers. Refer to Section 6.2.2.

14. Follow steps 4 through 7 listed in Section 6.3.

6.5.1 Sampling Using Stainless Steel Soil Corers

A soil corer is a stainless steel tube equipped with a cutting shoe and sample window in the side. The soil corer is advanced into the soil by applying downward pressure (body weight). The soil is unloaded by then forcing a ram towards the cutting shoe, which results in the discharge of the soil core through a window in the sleeve.

Use, application, and sample protocol is the same as for hand augering provided above, but without necessarily rotating the corer while advancing it.

SAFETY REMINDER

Hand augering and soil corer sampling can be physically demanding based on the type of geology and subsurface encumbrances encountered. Soil coring has some added hazards such the corer collapsing under your weight. To reduce the potential for muscle strain and damage, the following measures will be incorporated:

- Stretch and limber your muscles before heavy exertion. This hazard becomes more predominant in the early morning hours (prior to muscles becoming limber) and later in the day (as a result of fatigue).

- Job rotation – Share the duties so that repetitive actions do not result in fatigue and injury.

- Increase break frequencies as needed, especially as ambient conditions of heat and/or cold stress may dictate.

- Do not force the hand tools or use cheater pipes or similar devices to bypass an obstruction. Move to another location near the sampling point. Exerting additional forces on the sampling devices can result in damage and/or failure that could potentially injure someone in the immediate vicinity.

- Do not over compromise yourself when applying force to the soil corer or hand auger. If there is a sudden release, it could result in a fall or muscle injury due to strain.

6.6 Subsurface Soil Sampling with a Split-Barrel Sampler

A split-barrel (split-spoon) sampler consists of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-pound or larger casing driver.

Safety Reminder

It is intended through the Equipment Inspection for Drill Rigs form provided in the HASP that the hammer and hemp rope, where applicable, associated with this activity will be inspected (no physical damage is obvious), properly attached to the hammer (suitable knots or sufficient mechanical devices), and is in overall good condition.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (2-inch OD, 1-3/8-inch ID, either 20 inches or 26 inches long); Larger OD samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-pound weight, driving head, and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.
- Equipment listed in Section 6.3.

The following steps shall be followed to obtain split-barrel samples (Steps 1 through 4 are typically performed by the drilling subcontractor):

1. Attach the split-barrel sampler to the sampling rods.

2. Lower the sampler into the borehole inside the hollow stem auger bits.

3. Advance the split-barrel sampler by hammering the length (typically 18 or 24 inches) of the split-barrel sampler into the soil using 140-pound or larger hammer.

4. When the desired depth is achieved, extract the drill rods and sampler from the augers and/or borehole.

5. Detach the sampler from the drill rods.

6. Place the sampler securely in a vise so it can be opened using pipe wrenches.

> ***CAUTION***
> Pipe wrenches are used to separate the split spoon into several components. The driller's helper should not apply excessive force through the use of cheater pipes or push or pull in the direction where, if the wrench slips, hands or fingers will be trapped against an immovable object.

7. Remove the drive head and nosepiece with the wrenches, and open the sampler to reveal the soil sample.

8. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.) (as project-specific planning documents dictate). Carefully separate (or cut) the soil core, with a decontaminated stainless steel knife or trowel, at about 6-inch intervals while scanning the center of the core for elevated readings. Also scan stained soil, soil lenses, and anomalies (if present), and record readings.

9. If elevated vapor readings were observed, collect the sample scheduled for volatile analysis from the center of the core where elevated readings occurred. If no elevated readings where encountered, the sample material should be collected from the core's center (this area represents the least disturbed area with minimal atmospheric contact) (refer to Section 6.2.1).

10. Using the same trowel, remove remaining sample material from the split-barrel sampler (except for the small portion of disturbed soil usually found at the top of the core sample) and place the soil into a decontaminated stainless steel mixing bowl.

11. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers (refer to Section 6.2.2).

12. Follow steps 4 through 7 in Section 6.3.

6.7 Subsurface Soil Sampling Using Direct-Push Technology

Subsurface soil samples can be collected to depths of 40+ feet using DPT. DPT equipment, responsibilities, and procedures are described in SOP SA-2.5.

6.8 Excavation and Sampling of Test Pits and Trenches

6.8.1 Applicability

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

CAUTION

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise from the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden, steel, or aluminum support structures or through sloping and benching. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments; therefore, monitoring will be conducted by the Competent Person to determine if it is safe to enter. Any entry into a trench greater than 4 feet deep will constitute a Confined Space Entry and must be conducted in conformance with OSHA standard 29 CFR 1910.146. In all cases involving entry, substantial air monitoring, before entry, appropriate respiratory gear and protective clothing determination, and rescue provisions are mandatory. There must be at least three people present at the immediate site before entry by one of the field team members. This minimum number of people will increase based on the potential hazards or complexity of the work to be performed. The reader shall refer to OSHA regulations 29 CFR 1926.650, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146. High-hazard entries such as this will be supported by members of the Health Sciences Group professionally trained in these activities.

Excavations are generally not practical where a depth of more than about 15 to 20-feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If soil data at depths greater than 15-feet are required, the data are usually obtained through test borings instead of test pits.

In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations.

6.8.2 Test Pit and Trench Excavation

Test pits or trench excavations are constructed with the intent that they will provide an open view of subsurface lithology and/or disposal conditions that a boring will not provide. These procedures describe the methods for excavating and logging test pits and trenches installed to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).

Test pits and trenches may be excavated by hand or power equipment to permit detailed descriptions of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:

- The purpose and extent of the exploration

- The space required for efficient excavation
- The chemicals of concern
- The economics and efficiency of available equipment

Test pits normally have a cross section that is 4 to 10 feet square; test trenches are usually 3 to 6 feet wide and may be extended for any length required to reveal conditions along a specific line. The following table provides guidelines for design consideration based on equipment efficiencies.

Equipment	Typical Widths, in Feet
Trenching machine	0.25 to 1.0
Backhoe/Track Hoe	2 to 6

The lateral limits of excavation of trenches and the position of test pits shall be carefully marked on area base maps. If precise positioning is required to indicate the location of highly hazardous materials, nearby utilities, or dangerous conditions, the limits of the excavation shall be surveyed. Also, if precise determination of the depth of buried materials is needed for design or environmental assessment purposes, the elevation of the ground surface at the test pit or trench location shall also be determined by survey. If the test pit/trench will not be surveyed immediately, it shall be backfilled and its position identified with stakes placed in the ground at the margin of the excavation for later surveying.

The construction of test pits and trenches shall be planned and designed in advance as much as possible. However, the following field conditions may necessitate revisions to the initial plans:

- Subsurface utilities
- Surface and subsurface encumbrances
- Vehicle and pedestrian traffic patterns
- Purpose for excavation (e.g., the excavation of potential ordnance items)

The final depth and construction method shall be collectively determined by the FOL and designated Competent Person. The actual layout of each test pit, temporary staging area, and spoils pile may further be predicated based on site conditions and wind direction at the time the test pit is excavated. Prior to excavation, the area may be surveyed by magnetometer or metal detector or other passive methods specified in SOP HS1.0, Utility Location and Excavation Clearance, to identify the presence of underground utilities or drums. Where possible, the excavator should be positioned upwind and preferably within an enclosed cab.

No personnel shall enter any test pit or excavation except as a last resort, and then only under direct supervision of a Competent Person. If entrance is required, OSHA requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be placed for every 25 feet of lateral travel and extended 3 feet above ground surface). A temporary guard rail or vehicle stop must be placed along the surface of the hole before entry in situations where the excavation may be approached by traffic. Spoils will be stockpiled no closer than 2 feet from the sidewall of the excavation. The excavation equipment operator shall be careful not to undercut sidewalls and will, where necessary, bench back to increase stability. The top cover, when considered clean, will be placed separately from the subsurface materials to permit clean cover. It is emphasized that the project data needs should be structured such that required samples can be collected without requiring entrance into the excavation. For example,

samples of leachate, groundwater, or sidewall soil can be collected with telescoping poles or similar equipment.

Dewatering and watering may be required to ensure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation stable. This is an important consideration for excavations in cohesionless material below the groundwater table and for excavations left open greater than a day. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.

Where possible excavations and test pits shall be opened and closed within the same working day. Where this is not possible, the following engineering controls shall be put in place to control access:

- Trench covers/street plates
- Fences encompassing the entire excavation intended to control access
- Warning signs warning personnel of the hazards
- Amber flashing lights to demarcate boundaries of the excavation at night

Excavations left open will have emergency means to exit should someone accidentally enter.

6.8.3 Sampling in Test Pits and Trenches

6.8.3.1 General

Log test pits and trenches as they are excavated in accordance with the Test Pit Log presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.

Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable health and safety and OSHA requirements have been met as stated above. These provisions will be reiterated as appropriate in the project-specific HASP.

The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.

In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information includes soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples that can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost-effective method of sampling than installing borings.

6.8.3.2 Sampling Equipment

The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:

- Backhoe or other excavating machinery.
- Shovels, picks, hand augers, and stainless steel trowels/disposable trowels.
- Sample container - bucket with locking lid for large samples; appropriate bottle ware for chemical or geotechnical analysis samples.
- Polyethylene bags for enclosing sample containers; buckets.
- Remote sampler consisting of 10-foot sections of steel conduit (1-inch-diameter), hose clamps, and right angle adapter for conduit (see Attachment D).

6.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 6.8.3.4.

- Excavate the trench or pit in several 0.5- to 1.0-foot depth increments. Where soil types support the use of a sand bar cutting plate, use of this device is recommended to avoid potentially snagging utilities with the excavator teeth. It is recommended that soil probes or similar devices be employed where buried items or utilities may be encountered. This permits the trench floor to be probed prior to the next cut.
- After each increment:
  - the operator shall wait while the sampler inspects the test pit from grade level
  - the sampler shall probe the next interval where this is considered necessary. Practical depth increments for lithological evaluations may range from 2 to 4 feet i or where lithological changes are noted.
- The backhoe operator, who will have the best view of the test pit, shall immediately cease digging if:
  - Any fluid phase, including groundwater seepage, is encountered in the test pit
  - Any drums, other potential waste containers, obstructions, or utility lines are encountered
  - Distinct changes of material being excavated are encountered

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending on the conditions encountered, it may be required to excavate more slowly and carefully with the backhoe.

For obtaining test pit samples from grade level, the following procedure shall be followed:

- Use the backhoe to remove loose material from the excavation walls and floor to the greatest extent possible.
- Secure the walls of the pit, if necessary. (There is seldom any need to enter a pit or trench that would justify the expense of shoring the walls. All observations and samples should be taken from the ground surface.)

- Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material after it has been deposited on the ground, as follows:

  a. The sampler or FOL shall direct the backhoe operator to remove material from the selected depth or location within the test pit/trench.

  b. The backhoe operator shall bring the bucket over to a designated location on the sidewall a sufficient distance from the pit (at least 5 feet) to allow the sampler to work around the bucket.

  c. After the bucket has been set on the ground, the backhoe operator shall either disengage the controls or shut the machine down.

  d. When signaled by the operator that it is safe to do, the sampler will approach the bucket.

  e. The soil shall be monitored with a photoionization or flame ionization detector (PID or FID) as directed in the project -specific planning documents.

  f. The sampler shall collect the sample from the center of the bucket or pile in accordance with surface soil sampling procedures of Section 6.3 or 6.4, as applicable. Collecting samples from the center of a pile or bucket eliminates cross-contamination from the bucket or other depth intervals.

- If a composite sample is desired, several depths or locations within the pit/trench will be selected, and the bucket will be filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.

> ***CAUTION***
>
> Care must be exercised when using the remote sampler described in the next step because of potential instability of trench walls. In situations where someone must move closer than 2 feet to the excavation edge, a board or platform should be used to displace the sampler's weight to minimize the chance of collapse of the excavation edge. Fall protection should also be employed when working near the edges or trenches greater than 6 feet deep. An immediate means to extract people who have fallen into the trench will be immediately available. These means may include ladders or rope anchor points.

- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the sidewall or bottom of the pit as follows:

  a. Scrape the face of the pit/trench using a long-handled shovel or hoe to remove the smeared zone that has contacted the backhoe bucket.

  b. Collect the sample directly into the sample jar, by scraping with the jar edge, eliminating the need for sample handling equipment and minimizing the likelihood of cross-contamination.

  c. Cap the sample jar, remove it from the remote sampler assembly, and package the sample for shipment in accordance with SOP SA-6.3.

- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

6.8.3.4 In-Pit Sampling

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of soil or wastes within the test pit/trench) or when samples from relatively small discrete zones within the test pit are required. This approach may also be necessary to sample any seepage occurring at discrete levels or zones in the test pit that are not accessible with remote samplers.

In general, personnel shall sample and log pits and trenches from the ground surface, except as provided for by the following criteria:

- There are no practical alternative means of obtaining such data.
- The SSO and Competent Person determine that such action can be accomplished without breaching site safety protocol. This determination will be based on actual monitoring of the pit/trench after it is dug (including, at a minimum, measurements of oxygen concentration, flammable gases, and toxic compounds, in that order). Action levels will be provided in project-specific planning documents.
- A company-designated Competent Person determines that the pit/trench is stable trough soil classification evaluation/inspections or is made stable (by cutting/grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements shall be strictly observed.

If these conditions are satisfied, only one person may enter the pit/trench. On potentially hazardous waste sites, this individual shall be dressed in selected PPE as required by the conditions in the pit. He/she shall be affixed to a harness and lifeline and continuously monitored while in the pit.

A second and possible third individual shall be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations to support self rescue or assisted self rescue. The individual entering the pit shall remain therein for as brief a period as practical, commensurate with performance of his/her work. After removing the smeared zone, samples shall be obtained with a decontaminated trowel or spoon.

6.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 6.8.3.2, the following equipment is needed for geotechnical sampling:

- Soil sampling equipment, similar to that used in shallow drilled boring (i.e., thin-walled tube samplers), that can be pushed or driven into the floor of the test pit.
- Suitable driving (e,g., sledge hammer) or pushing (e.g., backhoe bucket) equipment used to advance the sampler into the soil.
- Knives, spatulas, and other suitable devices for trimming hand-carved samples.
- Suitable containers (bags, jars, tubes, boxes, etc.), labels, wax, etc. for holding and safely transporting collected soil samples.
- Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

Disturbed grab or bulk geotechnical soil samples may be collected for most soil in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic-lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification: larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soil using thin-walled tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability, and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe, rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the tube when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 6.8.3.4 shall be followed. The thin-walled tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate because the sample will not have the correct orientation.

A sledge hammer or backhoe may be used to drive or push the tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. When using a sledge hammer, it is recommended that the sampler be stabilized using a rope/strap wrench or pipe wrench to remove the person's hands holding the sampler from the strike zone. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hook the sampler to the excavator or backhoe and extract. This means an alternative head will be used as a connection point or that multiple choke hitches will be applied to extract the sampler. If this fails and the excavator can dig deeper without potentially impacting subsurface utilities, excavate the sampler. If this fails or if the excavator cannot be used due to subsurface utilities, hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry into the test pit, the requirements in Section 6.8.3.4 must be followed. Prepare the sample as described in Steps 9 through 13 in Section 6.2.3, and label, pack and transport the sample in the required manner, as described in SOPs SA-6.3 and SA-6.1.

6.8.4 Backfilling of Trenches and Test Pits

All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.

Before backfilling, the onsite crew may photograph, if required by the project-specific work plan, all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.

After inspection, backfill material shall be returned to the pit under the direction of the FOL. Backfill should be returned to the trench or test pit in 6-inch to 1-foot lifts and compacted with the bucket. Remote controlled tampers or rollers may be lowered into the trench and operated from top side. This procedure will continue to the grade surface. It is recommended that the trench be tracked or rolled in. During excavation, clean soil from the top 2 feet may have been separated to be used to cover the last segments. Where these materials are not clean, it is recommended that clean fill be used for the top cover.

If a low-permeability layer is penetrated (resulting in groundwater flow from an upper contaminated flow zone into a lower uncontaminated flow zone), backfill material must represent original conditions or be impermeable. Backfill could consist of a soil-bentonite mix prepared in a proportion specified by the FOL (representing a permeability equal to or less than original conditions). Backfill can be covered by "clean" soil and graded to the original land contour. Revegetation of the disturbed area may also be required.

6.9 Records

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler for all samples collected. All soil sampling locations should be documented by tying in the location of two or more nearby permanent landmarks (building, telephone pole, fence, etc.) or obtaining GPS coordinates; and shall be noted on the appropriate sample log sheet, site map, or field notebook. Surveying may also be necessary, depending on the project requirements.

Test pit logs (see Attachment C of this SOP) shall contain a sketch of pit conditions. If the project-specific work plan requires photographs, at least one photograph with a scale for comparison shall be taken of each pit. Included in the photograph shall be a card showing the test pit number. Boreholes, test pits, and trenches shall be logged by the field geologist in accordance with SOP GH-1.5.

Other data to be recorded in the field logbook include the following:

- Name and location of job
- Date of boring and excavation
- Approximate surface elevation
- Total depth of boring and excavation
- Dimensions of pit
- Method of sample acquisition
- Type and size of samples
- Soil and rock descriptions
- Photographs if required
- Groundwater levels
- PID/FID/LEL/O_2 meter readings
- Other pertinent information, such as waste material encountered

In addition, site-specific documentation to be maintained by the SSO and/or Competent Person will be required including:

- Calibration logs
- Excavation inspection checklists

- Soil type classification

7.0 REFERENCES

American Society for Testing and Materials, 1987. ASTM Standards D1587-83 and D1586-84. ASTM Annual Book of Standards. ASTM. Philadelphia, Pennsylvania. Volume 4.08.

NUS Corporation, 1986. Hazardous Material Handling Training Manual.

NUS Corporation and CH2M Hill, August, 1987. Compendium of Field Operation Methods. Prepared for the U.S. EPA.

OSHA, Excavation, Trenching and Shoring 29 CFR 1926.650-653.

OSHA, Confined Space Entry 29 CFR 1910.146.

USEPA, November 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.

ATTACHMENT A
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date: / Time: / Method: / Monitor Reading (ppm):	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)

COMPOSITE SAMPLE DATA:

Date: / Method: / Monitor Readings (Range in ppm):	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

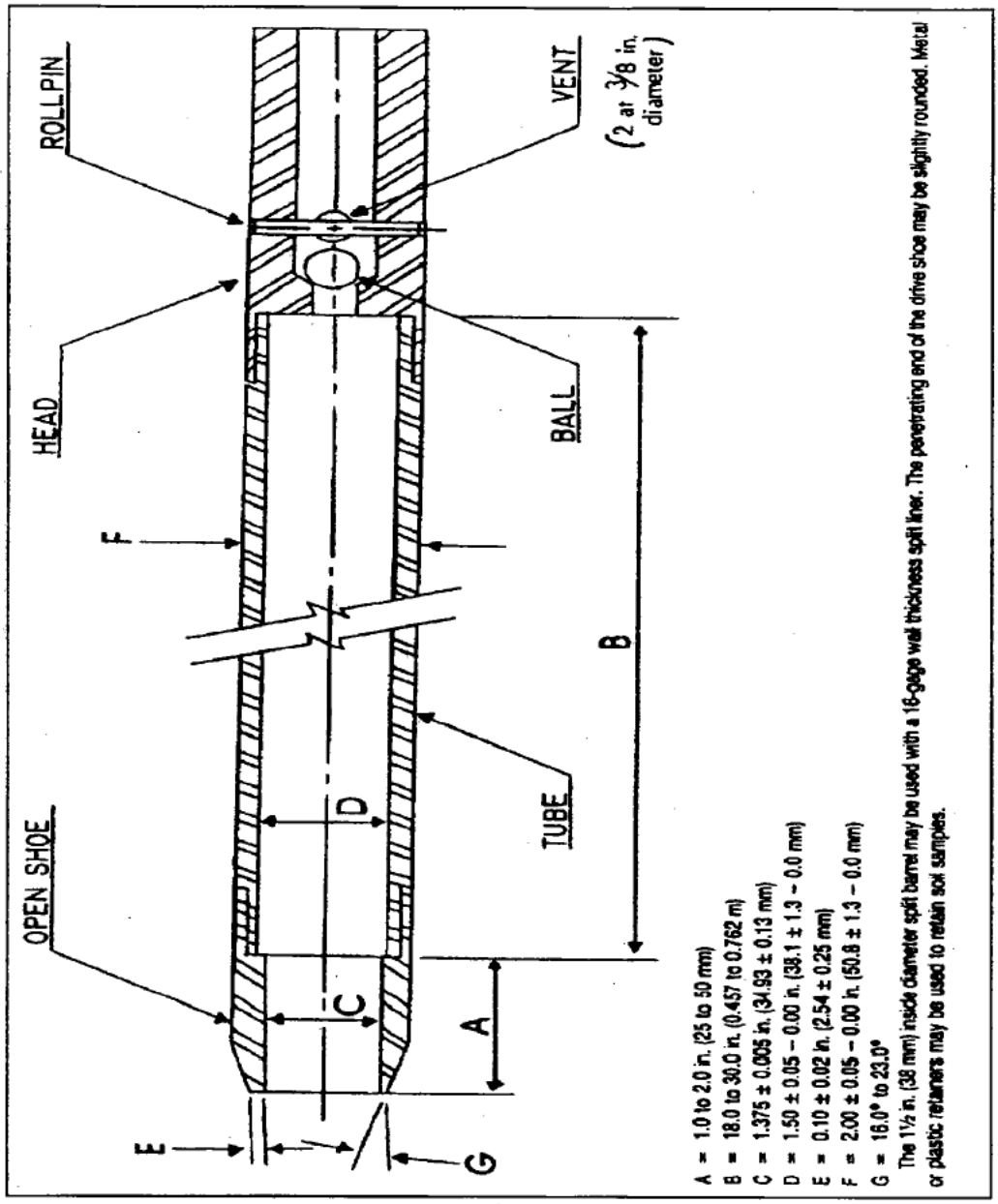
Circle if Applicable:

MS/MSD

Duplicate ID No.:

Signature(s):

ATTACHMENT B
SPLIT-SPOON SAMPLER



ATTACHMENT C
TEST PIT LOG

TEST PIT LOG Page ___ of ___

Tetra Tech NUS, Inc.

PROJECT NAME: _____ TEST PIT No.: _____
PROJECT NUMBER: _____ DATE: _____
LOCATION: _____ GEOLOGIST: _____

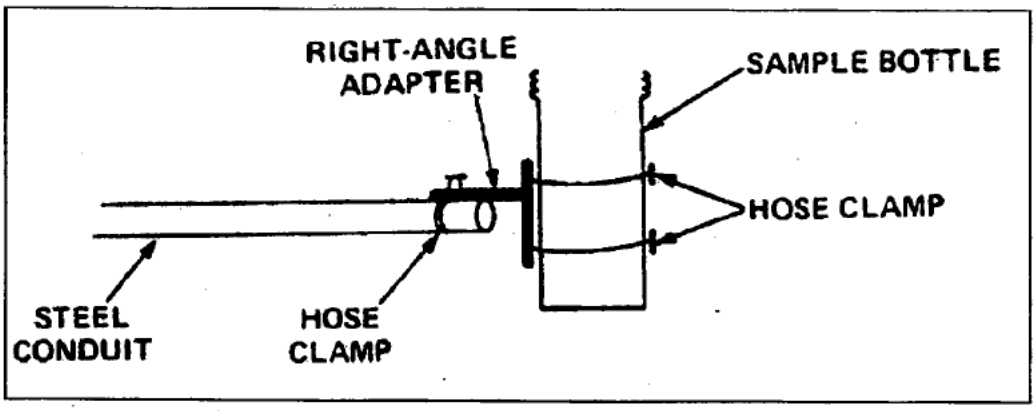
Depth (Ft.)	Lithology Change (Depth/Ft.)	MATERIAL DESCRIPTION: Soil/Waste Characteristics (lithology, density, color, etc.)	USCS	Remarks	PID/FID READING: Source (ppm)	PID/FID READING: BZ (ppm)

TEST PIT CROSS SECTION AND / OR PLAN VIEW

REMARKS: _____

PHOTO LOG: _____

ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING



TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number SA-6.3	Page 1 of 12
		Effective Date 03/09/09	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject FIELD DOCUMENTATION		Approved Tom Johnston	

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE 2

2.0 SCOPE 2

3.0 GLOSSARY 2

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS 2

5.0 PROCEDURES 2

5.1 SITE LOGBOOK 2
5.1.1 General 2
5.1.2 Photographs 3
5.2 FIELD NOTEBOOKS 3
5.3 FIELD FORMS 4
5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results. 4
5.3.2 Hydrogeological and Geotechnical Forms 5
5.3.3 Equipment Calibration and Maintenance Form 6
5.4 FIELD REPORTS 6
5.4.1 Daily Activities Report 7
5.4.2 Weekly Status Reports 7

6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE 7

ATTACHMENTS

A TYPICAL SITE LOGBOOK ENTRY 9
B SAMPLE LABEL 10
C CHAIN-OF-CUSTODY RECORD FORM 11
D CHAIN-OF-CUSTODY SEAL 12

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs, and reports generally initiated and maintained for documenting Tetra Tech NUS, Inc. (TtNUS) field activities.

2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM is responsible for obtaining hardbound controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The FOL is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports included in this SOP (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time frame.

General personnel qualifications for field documentation activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 SITE LOGBOOK

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2) but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

5.1.2 Photographs

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks into the project's central file.

5.2 FIELD NOTEBOOKS

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a

separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 FIELD FORMS

All TtNUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs, subject to client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOPs.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample log sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. Complete a sample log sheet for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record

The chain-of-custody record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc®-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The custody seal is an adhesive-backed label that is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. Sign and date custody seals

and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Complete Field Analytical Log Sheets to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

Complete a Groundwater Level Measurement Sheet for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.

5.3.2.3 Packer Test Report Form

Complete a Packer Test Report Form for each well at which a packer test is conducted.

5.3.2.4 Boring Log

Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

Complete a Monitoring Well Construction Details Form for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

5.3.2.7 Miscellaneous Monitoring Well Forms

Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.

5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.

- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.

- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.

- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring, or test equipment is necessary to ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log, which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

5.4 FIELD REPORTS

The primary means of recording on-site activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation but are not easily used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

5.4.1 Daily Activities Report

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

5.4.1.1 Description

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

In addition to those described herein, other summary reports may also be contractually required.

All TtNUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE

- Boring Log
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Hydraulic Conductivity Test Data Sheet
- Low Flow Purge Data Sheet
- Bedrock Monitoring Well Construction (Stick Up)
- Bedrock Monitoring Well Construction Flush Mount
- Bedrock Monitoring Well Construction Open Hole
- Confining Layer Monitoring Well Construction
- Monitoring Well Development Record

- Monitoring Well Materials Certificate of Conformance
- Overburden Monitoring Well Construction Flush Mount
- Overburden Monitoring Well Construction Stick Up
- Packer Test Report Form
- Pumping Test Data Sheet
- QA Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Surface Water Sample Log Sheet
- Test Pit Log
- Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____
PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

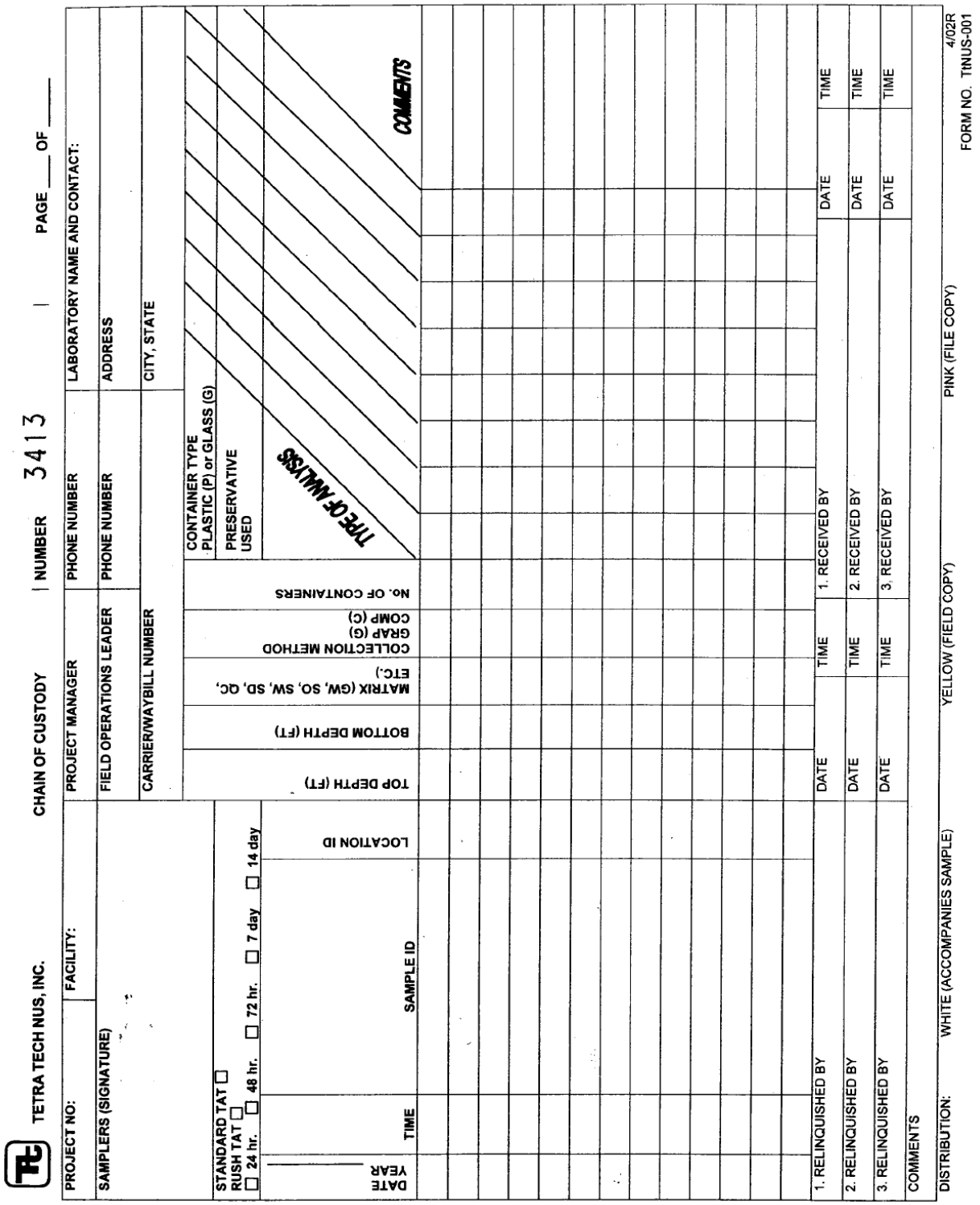
_____

Field Operations Leader

ATTACHMENT B
SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project: Site: Location:	
Sample No:			Matrix:
Date:	Time:	Preserve:	
Analysis:			
Sampled by:		Laboratory:	

ATTACHMENT C
CHAIN-OF-CUSTODY RECORD FORM



TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF ___

PROJECT NO: | FACILITY: | PROJECT MANAGER | PHONE NUMBER | LABORATORY NAME AND CONTACT:

SAMPLERS (SIGNATURE) | FIELD OPERATIONS LEADER | PHONE NUMBER | ADDRESS

CARRIER/WAYBILL NUMBER | CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐ ☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

DATE / YEAR / TIME | SAMPLE ID | LOCATION ID | TOP DEPTH (FT) | BOTTOM DEPTH (FT) | MATRIX (GW, SO, SW, SD, QC, ETC.) | COLLECTION METHOD GRAP (G) COMP (C) | No. OF CONTAINERS | CONTAINER TYPE PLASTIC (P) or GLASS (G) | PRESERVATIVE USED | TYPE OF ANALYSIS | COMMENTS

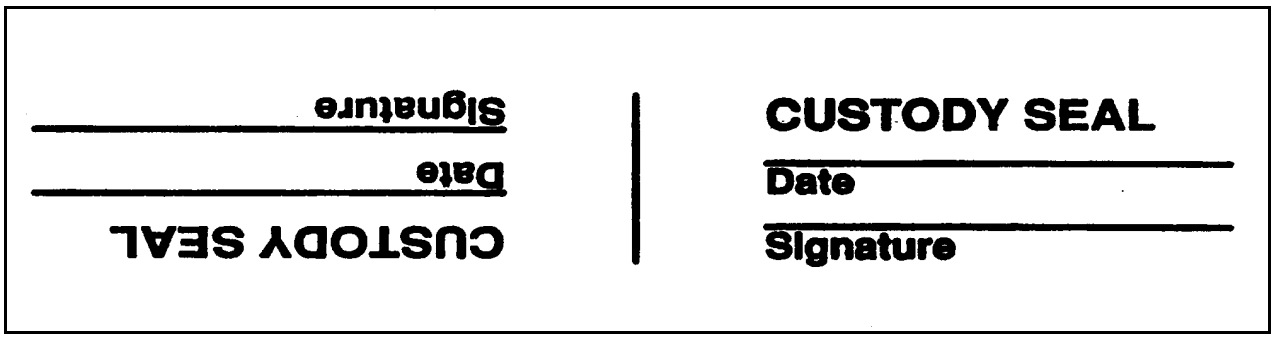
1. RELINQUISHED BY | DATE | TIME | 1. RECEIVED BY | DATE | TIME
2. RELINQUISHED BY | DATE | TIME | 2. RECEIVED BY | DATE | TIME
3. RELINQUISHED BY | DATE | TIME | 3. RECEIVED BY | DATE | TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

FORM NO. TtNUS-001 4/02R

ATTACHMENT D
CHAIN-OF-CUSTODY SEAL



TETRA TECH	STANDARD OPERATING PROCEDURES	Number SA-7.1	Page 1 of 16
		Effective Date 01/28/2009	Revision 6
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject DECONTAMINATION OF FIELD EQUIPMENT		Approved Tom Johnston	

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE 2

2.0 SCOPE AND APPLICABILITY 2

3.0 GLOSSARY 2

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS 3

5.0 HEALTH AND SAFETY 3

6.0 EQUIPMENT LIST 3

7.0 PROCEDURES 4

7.1 Decontamination Pad Design/Construction Considerations 5
7.1.1 Temporary Decontamination Pads 5
7.1.2 Decontamination Activities at Drill Rigs/DPT Units 7
7.1.3 Decontamination Activities at Remote Sample Locations 7
7.2 Equipment Decontamination Procedures 7
7.2.1 Monitoring Well Sampling Equipment 7
7.2.2 Downhole Drilling Equipment 9
7.2.3 Soil/Sediment Sampling Equipment 11
7.3 Contact Waste/Materials 11
7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments 12
7.4 Decontamination Evaluation 13

ATTACHMENTS

A INVESTIGATION-DERIVED WASTE LABEL 15

1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment , chemical sampling equipment and field operation and analytical equipment.

2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent – A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate decontamination procedures.

5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

7.1 Decontamination Pad Design/Construction Considerations

7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:

  - Well removed from pedestrian/vehicle thoroughfares.
  - Avoidance of areas where control/custody cannot be maintained.
  - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.
  - Avoidance of potentially contaminated areas.
  - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

  The selected decontamination site should include the following, where possible:

  - Areas where potable water and electricity are provided.

> ***Safety Reminder***
>
> When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:
>
> - All power is routed through a Ground Fault Circuit Interrupter (GFCI).
>
> - All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.
>
> In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.

- Areas that offer sufficient size to carry out the specific decontamination sequence.

- Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:

  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.

  - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.

  - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.

  - Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to provide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner, a light coating of sand shall be applied to provide traction as necessary.

  - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:

  - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.

- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

7.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

7.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

7.2.1 Monitoring Well Sampling Equipment

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.

2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.

3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.

4. Remove the pump and tubing/bailer from the container

5. Rinse external pump components using tap water.

6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

CAUTION

Do not rinse PE, PVC, and associated tubing with solvents –
Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.

8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.

9. Drain residual deionized water to the extent possible.

10. Allow components of the equipment to air dry.

11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.

12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

SAFETY REMINDER

Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

1. Wash with soap and water
2. Rinse with tap water
3. Rinse with deionized water

NOTE

In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.

- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:

  1. Wash with soap and water
  2. Rinse with tap water
  3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

7.2.2 Downhole Drilling Equipment

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

CAUTION

Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

CAUTION

In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol

5. To the extent possible, allow components to air dry.

6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.

7. Wrap or cover equipment in clear plastic until it is time to be used.

SAFETY REMINDER

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 ˚F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0˚ tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40˚ fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.

2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.

3. Always wear PPE as specified in the HASP such as:

   - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.

4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.

5. Do not modify equipment unless the manufacturer has approved the modifications.

7.2.3 Soil/Sediment Sampling Equipment

This section applies to soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1. Remove all loose soil from the equipment through manual means.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.

3. Rinse the equipment with tap water.

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.

5. Rinse the equipment with deionized water.

6. To the extent possible, allow components to air dry.

7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.

8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device

- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.

- Extract the dredge and wash it in soap and water per the project-specific planning documents.

> ***CAUTION***
> When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

> ***NOTE***
> Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

> ***NOTE***
> Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).

4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:

   - Enclose areas accessible by the general public using construction fencing and signs.
   - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.
   - Maintain the retaining bolt and label on the outside of storage containers where readily visible.
   - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.
   - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.
   - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

CAUTION

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

7.4 Decontamination Evaluation

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.

- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

NOTE

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:

  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.

  - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.

  - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:

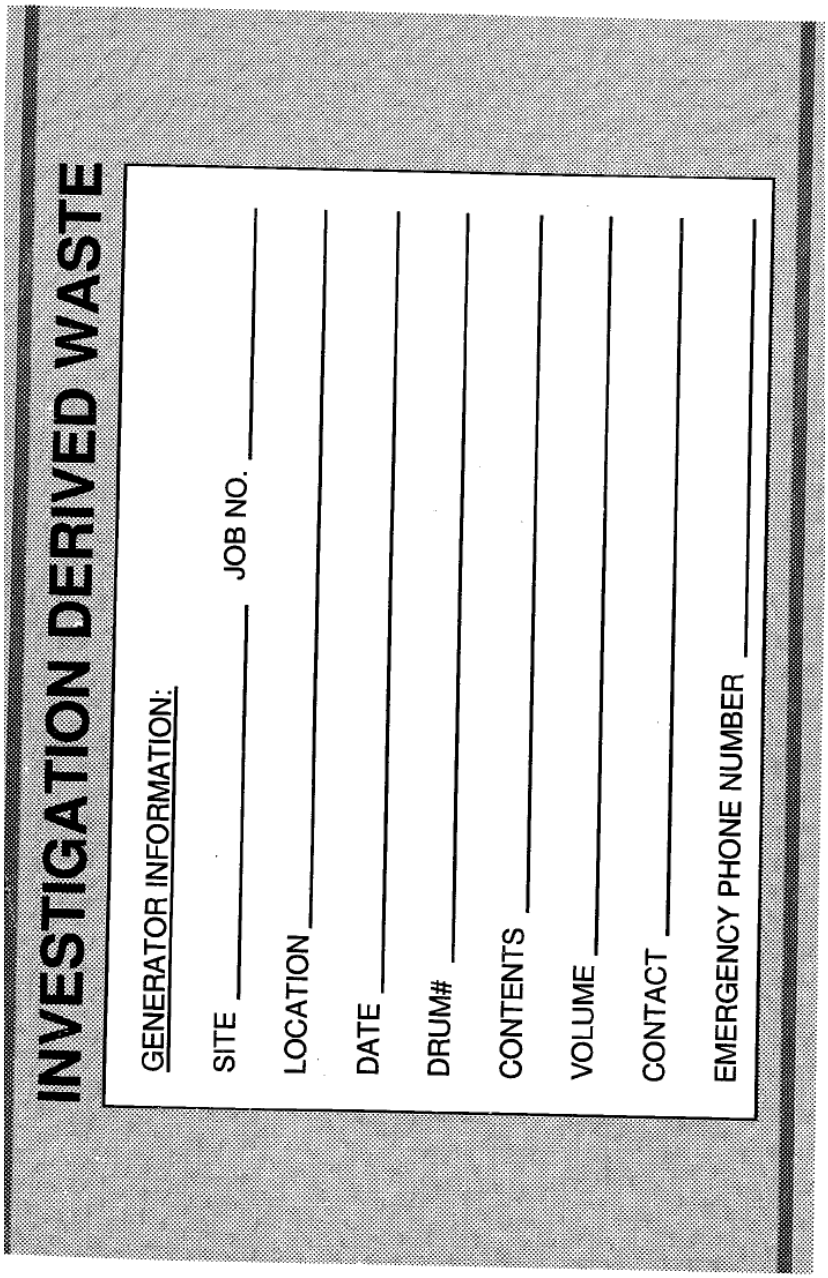
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

NOTE

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

Attachment A
iDW Label



TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number GH-1.3	Page 1 of 26
		Effective Date 06/99	Revision 1
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SOIL AND ROCK DRILLING METHODS		Approved D. Senovich	

TABLE OF CONTENTS

SECTION		PAGE
1.0	**PURPOSE**	**3**
2.0	**SCOPE**	**3**
3.0	**GLOSSARY**	**3**
4.0	**RESPONSIBILITIES**	**3**
5.0	**PROCEDURES**	**4**
5.1	GENERAL	4
5.2	DRILLING METHODS	4
5.2.1	Continuous-Flight Hollow-Stem Auger Drilling	5
5.2.2	Continuous-Flight Solid-Stem Auger Drilling	6
5.2.3	Rotary Drilling	7
5.2.4	Rotosonic Drilling	9
5.2.5	Reverse Circulation Rotary Drilling	9
5.2.6	Drill-through Casing Driver	10
5.2.7	Cable Tool Drilling	11
5.2.8	Jet Drilling (Washing)	12
5.2.9	Drilling with a Hand Auger	13
5.2.10	Rock Drilling and Coring	13
5.2.11	Drilling & Support Vehicles	14
5.2.12	Equipment Sizes	15
5.2.13	Estimated Drilling Progress	16
5.3	PREVENTION OF CROSS-CONTAMINATION	16
5.4	CLEANOUT OF CASING PRIOR TO SAMPLING	17
5.5	MATERIALS OF CONSTRUCTION	18
5.6	SUBSURFACE SOIL SAMPLES	19
5.7	ROCK SAMPLING (CORING) (ASTM D2113-83)	19
5.7.1	Diamond Core Drilling	23
5.7.2	Rock Sample Preparation and Documentation	23
6.0	**REFERENCES**	**24**

ATTACHMENT

A	DRILLING EQUIPMENT SIZES	25

FIGURE

NUMBER **PAGE**

1 STANDARD SIZES OF CORE BARRELS AND CASING 20

1.0 PURPOSE

The purpose of this procedure is to describe the methods and equipment necessary to perform soil and rock borings and identify the equipment, sequence of events, and appropriate methods necessary to obtain soil, both surface and subsurface, and rock samples during field sampling activities.

2.0 SCOPE

This guideline addresses most of the accepted and standard drilling techniques, their benefits, and drawbacks. It should be used generally to determine what type of drilling techniques would be most successful depending on site-specific geologic conditions and the type of sampling required.

The sampling methods described within this procedure are applicable to collecting surface and subsurface soil samples, and obtaining rock core samples for lithologic and hydrogeologic evaluation, excavation/foundation design, remedial alternative design and related civil engineering purposes.

3.0 GLOSSARY

Rock Coring - A method in which a continuous solid cylindrical sample of rock or compact rock-like soil is obtained by the use of a double tube core barrel that is equipped with an appropriate diamond-studded drill bit which is advanced with a hydraulic rotary drilling machine.

Wire-Line Coring - As an alternative to conventional coring, this technique is valuable in deep hole drilling, since this method eliminates trips in and out of the hole with the coring equipment. With this technique, the core barrel becomes an integral part of the drill rod string. The drill rod serves as both a coring device and casing.

4.0 RESPONSIBILITIES

Project Manager - In consultation with the project geologist, the Project Manager is responsible for evaluating the drilling requirements for the site and specifying drilling techniques that will be successful given the study objectives and the known or suspected geologic conditions at the site. The Project Manager also determines the disposal methods for products generated by drilling, such as drill cuttings and well development water, as well as any specialized supplies or logistical support required for the drilling operations.

Field Operations Leader (FOL) - The FOL is responsible for the overall supervision and scheduling of drilling activities, and is strongly supported by the project geologist.

Project Geologist - The project geologist is responsible for ensuring that standard and approved drilling procedures are followed. The geologist will generate a detailed boring log for each test hole. This log shall include a description of materials, samples, method of sampling, blow counts, and other pertinent drilling and testing information that may be obtained during drilling (see SOPs SA-6.3 and GH-1.5). Often this position for inspecting the drilling operations may be filled by other geotechnical personnel, such as soils and foundation engineers, civil engineers, etc.

Determination of the exact location for borings is the responsibility of the site geologist. The final location for drilling must be properly documented on the boring log. The general area in which the borings are to be located will be shown on a site map included in the Work Plan and/or Sampling and Analysis Plan.

Drilling Subcontractor - Operates under the supervision of the FOL. Responsible for obtaining all drilling permits and clearances, and supplying all services (including labor), equipment and material required to perform the drilling, testing, and well installation program, as well as maintenance and quality control of such required equipment except as stated in signed and approved subcontracts.

The driller must report any major technical or analytical problems encountered in the field to the FOL within 24 hours of determination, and must provide advance written notification of any changes in field procedures, describing and justifying such changes. No such changes shall be made unless requested and authorized in writing by the FOL (with the concurrence of the Project Manager). Depending on the subcontract, the Project Manager may need to obtain written authorization from appropriate administrative personnel before approving any changes.

The drilling subcontractor is responsible for following decontamination procedures specified in the project plan documents. Upon completion of the work, the driller is responsible for demobilizing all equipment, cleaning up any materials deposited on site during drilling operations, and properly backfilling any open borings.

5.0 PROCEDURES

5.1 General

The purpose of drilling boreholes is:

- To determine the type, thickness, and certain physical and chemical properties of the soil, water and rock strata which underlie the site.
- To install monitoring wells or piezometers.

All drilling and sampling equipment will be cleaned between samples and borings using appropriate decontamination procedures as outlined in SOP SA-7.1. Unless otherwise specified, it is generally advisable to drill borings at "clean" locations first, and at the most contaminated locations last, to reduce the risk of spreading contamination between locations. All borings must be logged by the site geologist as they proceed (see SOPs SA-6.3 and GH-1.5). Situations where logging would not be required would include installation of multiple well points within a small area, or a "second attempt" boring adjacent to a boring that could not be continued through resistant material. In the latter case, the boring log can be resumed 5 feet above the depth at which the initial boring was abandoned, although the site geologist should still confirm that the stratigraphy at the redrilled location conforms essentially with that encountered at the original location. If significant differences are seen, each hole should be logged separately.

5.2 Drilling Methods

The selected drilling methods described below apply to drilling in subsurface materials, including, but not limited to, sand, gravel, clay, silt, cobbles, boulders, rock and man-made fill. Drilling methods should be selected after studying the site geology and terrain, the waste conditions at the site, and reviewing the purpose of drilling and the overall subsurface investigation program proposed for the site. The full range of different drilling methods applicable to the proposed program should be identified with final selection based on relative cost, availability, time constraints, and how well each method meets the sampling and testing requirements of the individual drilling program.

5.2.1 Continuous-Flight Hollow-Stem Auger Drilling

This method of drilling consists of rotating augers with a hollow stem into the ground. Cuttings are brought to the surface by the rotating action of the auger. This method is relatively quick and inexpensive. Advantages of this type of drilling include:

- Samples can be obtained without pulling the augers out of the hole. However, this is a poor method for obtaining grab samples from thin, discrete formations because of mixing of soils which occurs as the material is brought to the surface. Sampling of such formations requires the use of split-barrel or thin-wall tube samplers advanced through the hollow core of the auger.
- No drilling fluids are required.
- A well can be installed inside the auger stem and backfilled as the augers are withdrawn.

Disadvantages and limitations of this method of drilling include:

- Augering can only be done in unconsolidated materials.

- The inside diameter of hollow stem augers used for well installation should be at least 4 inches greater than the well casing. Use of such large-diameter hollow-stem augers is more expensive than the use of small-diameter augers in boreholes not used for well installation. Furthermore, the density of unconsolidated materials and depths become more of a limiting factor. More friction is produced with the larger diameter auger and subsequently greater torque is needed to advance the boring.

- The maximum effective depth for drilling is 150 feet or less, depending on site conditions and the size of augers used.

- In augering through clean sand formations below the water table, the sand will tend to flow into the hollow stem when the plug is removed for soil sampling or well installation. If the condition of "running" or "flowing" sands is persistent at a site, an alternative method of drilling is recommended, in particular for wells or boreholes deeper than 25 feet.

Hollow-stem auger drilling is the preferred method of drilling. Most alternative methods require the introduction of water or mud downhole (air rotary is the exception) to maintain the open borehole. With these other methods, great care must be taken to ensure that the method does not interfere with the collection of a representative sample (which may be the prime objective of the borehole construction). With this in mind, the preferred order of choice of drilling method after hollow-stem augering (HSA) is:

- Cable tool
- Casing drive (air)
- Air rotary
- Mud rotary
- Rotosonic
- Drive and wash
- Jetting

However, the use of any method will also depend on efficiency and cost-effectiveness. In many cases, mud rotary is the only feasible alternative to hollow-stem augering. Thus, mud rotary drilling is generally acceptable as a first substitute for HSA.

The procedures for sampling soils through holes drilled by hollow-stem auger shall conform with the applicable ASTM Standards: D1587-83 and D1586-84. The guidelines established in SOP SA-1.3 shall

also be followed. The hollow-stem auger may be advanced by any power-operated drilling machine having sufficient torque and ram range to rotate and force the auger to the desired depth. The machine must, however, be equipped with the accessory equipment needed to perform required sampling, or rock coring.

The hollow-stem auger may be used without the plug when boring for geotechnical examination or for well installation. However, when drilling below the water table, specially designed plugs which allow passage of formation water but not solid material shall be used (see Reference 1 of this guideline). This drilling configuration method also prevents blow back and plugging of the auger when the plug is removed for sampling.

Alternately, it may be necessary to keep the hollow stem full of water, at least to the level of the water table, to prevent blowback and plugging of the auger. If water is added to the hole, it must be sampled and analyzed to determine if it is free from contaminants prior to use. In addition, the amount of water introduced, the amount recovered upon attainment of depth, and the amount of water extracted during well development must be carefully logged in order to ensure that a representative sample of the formation water can be obtained. Well development should occur as soon after well completion as practicable (see SOP GH-2.8 for well development procedures). If gravelly or hard material is encountered which prevents advancing the auger to the desired depth, augering should be halted and either driven casing or hydraulic rotary methods should be attempted. If the depth to the bedrock/soil interface and bedrock lithology must be determined, then a 5-foot confirmatory core run should be conducted (see Section 5.2.9).

At the option of the Field Operations Leader (in communication with the Project Manager), when resistant materials prevent the advancement of the auger, a new boring can be attempted. The original boring must be properly backfilled and the new boring started a short distance away at a location determined by the site geologist. If multiple water bearing strata were encountered, the original boring must be grouted. In some formations, it may be prudent to also grout borings which penetrate only the water table aquifer, since loose soil backfill in the boring may still provide a preferred pathway for surface liquids to reach the water table. Backfilling requirements may also be driven by state or local regulations.

5.2.2 Continuous-Flight Solid-Stem Auger Drilling

This drilling method is similar to hollow-stem augering. Practical application of this method is severely restricted compared to use of hollow-stem augers. Split-barrel (split-spoon) sampling cannot be performed without pulling the augers out, which may allow the hole to collapse. The continuous-flight solid-stem auger drilling method is therefore very time consuming and is not cost effective. Also, augers would have to be withdrawn before installing a monitoring well, which again, may allow the hole to collapse. Furthermore, geologic logging by examining the soils brought to the surface is unreliable, and depth to water may be difficult to determine while drilling.

There would be very few situations where use of a solid-stem auger would be preferable to other drilling methods. The only practical applications of this method would be to drill boreholes for well installation where no lithologic information is desired and the soils are such that the borehole can be expected to remain open after the augers are withdrawn. Alternatively, this technique can be used to find depth to bedrock in an area when no other information is required from drilling.

5.2.3 Rotary Drilling

Direct rotary drilling includes air-rotary and fluid-rotary drilling. For air or fluid-rotary drilling, the rotary drill may be advanced to the desired depth by any power-operated drilling machine having sufficient torque

and ram range to rotate and force the bit to the desired depth. The drilling machine must, however, be equipped with any accessory equipment needed to perform required sampling, or coring. Prior to sampling, any settled drill cuttings in the borehole must be removed.

Air-rotary drilling is a method of drilling where the drill rig simultaneously turns and exerts a downward pressure on the drilling rods and bit while circulating compressed air down the inside of the drill rods, around the bit, and out the annulus of the borehole. Air circulation serves to both cool the bit and remove the cuttings from the borehole. Advantages of this method include:

- The drilling rate is high (even in rock).
- The cost per foot of drilling is relatively low.
- Air-rotary rigs are common in most areas.
- No drilling fluid is required (except when water is injected to keep down dust).
- The borehole diameter is large, to allow room for proper well installation procedures.

Disadvantages to using this method include:

- Formations must be logged from the cuttings that are blown to the surface and thus the depths of materials logged are approximate.
- Air blown into the formation during drilling may "bind" the formation and impede well development and natural groundwater flow.
- In-situ samples cannot be taken, unless the hole is cased.
- Casing must generally be used in unconsolidated materials.
- Air-rotary drill rigs are large and heavy.
- Large amounts of Investigation Derived Waste (IDW) may be generated which may require containerization, sampling, and off-site disposal.

A variation of the typical air-rotary drill bit is a down hole hammer which hammers the drill bit down as it drills. This makes drilling in hard rock faster. Air-rotary drills can also be adapted to use for rock coring although they are generally slower than other types of core drills. A major application of the air-rotary drilling method would be to drill holes in rock for well installation.

Fluid-Rotary drilling operates in a similar manner to air-rotary drilling except that a drilling fluid ("mud") or clean water is used in place of air to cool the drill bit and remove cuttings. There are a variety of fluids that can be used with this drilling method, including bentonite slurry and synthetic slurries. If a drilling fluid other than water/cuttings is used, it must be a natural clay (i.e., bentonite) and a "background" sample of the fluid should be taken for analysis of possible organic or inorganic contaminants.

Advantages to the fluid-rotary drilling method include:

- The ability to drill in many types of formations.
- Relatively quick and inexpensive.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be obtained without removing drill rods if the appropriate size drill rods and bits (i.e., fish-tail or drag bit) are used.

- In some borings temporary casing may not be needed as the drilling fluids may keep the borehole open.
- Drill rigs are readily available in most areas.

Disadvantages to this method include:

- Formation logging is not as accurate as with hollow-stem auger method if split-barrel (split-spoon) samples are not taken (i.e., the depths of materials logged from cuttings delivered to the surface are approximate).
- Drilling fluids reduce permeability of the formation adjacent to the boring to some degree, and require more extensive well development than "dry" techniques (augering, air-rotary).
- No information on depth to water is obtainable while drilling.
- Fluids are needed for drilling, and there is some question about the effects of the drilling fluids on subsequent water samples obtained. For this reason as well, extensive well development may be required.
- In very porous materials (i.e., rubble fill, boulders, coarse gravel) drilling fluids may be continuously lost into the formation. This requires either constant replenishment of the drilling fluid, or the use of casing through this formation.
- Drill rigs are large and heavy, and must be supported with supplied water.
- Groundwater samples can be potentially diluted with drilling fluid.

The procedures for performing direct rotary soil investigations and sampling shall conform with the applicable ASTM standards: D2113-83, D1587-83, and D1586-84.

Soil samples shall be taken as specified by project plan documents, or more frequently, if requested by the project geologist. Any required sampling shall be performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool.

When field conditions prevent the advancement of the hole to the desired depth, a new boring may be drilled at the request of the Field Operations Leader. The original boring shall be backfilled using methods and materials appropriate for the given site and a new boring started a short distance away at a location determined by the project geologist.

5.2.4 Rotosonic Drilling

The Rotosonic drilling method employs a high frequency vibrational and low speed rotational motion coupled with down pressure to advance the cutting edge of a drill string. This produces a uniform borehole while providing a continuous, undisturbed core sample of both unconsolidated and most bedrock formations. Rotosonic drilling advances a 4-inch diameter to 12-inch diameter core barrel for sampling and can advance up to a 12-inch diameter outer casing for the construction of standard and telescoped monitoring wells. During drilling, the core barrel is advanced ahead of the outer barrel in increments as determined by the site geologist and depending upon type of material, degree of subsurface contamination and sampling objectives.

The outer casing can be advanced at the same time as the inner drill string and core barrel, or advanced down over the inner drill rods and core barrel, or after the core barrel has moved ahead to collect the undisturbed sample and has been pulled out of the borehole. The outer casing can be advanced dry in most cases, or can be advanced with water or air depending upon the formations being drilled, the depth and diameter of the hole, or requirements of the project.

Advantages of this method include:

- Sampling and well installation are faster as compared to other drilling methods.
- Continuous sampling, with larger sample volume as compared to split-spoon sampling.
- The ability to drill through difficult formations such as cobbles or boulders, hard till and bedrock.
- Reduction of IDW by an average of 70 to 80 percent.
- Well installations are quick and controlled by elimination of potential bridging of annular materials during well installation, due to the ability to vibrate the outer casing during removal.

Disadvantages include:

- The cost for Rotosonic drilling as compared to other methods are generally higher. However, the net result can be a significant savings considering reduced IDW and shortened project duration.
- Rotosonic drill rigs are large and need ample room to drill, however, Rotosonic units can be placed on the ground or placed on an ATV.
- There are a limited number of Rotosonic drilling contractors at the present time.

5.2.5 Reverse Circulation Rotary Drilling

The common reverse-circulation rig is a water or mud-rotary rig with a large-diameter drill pipe which circulates the drilling water down the annulus and up the inside of the drill pipe (reverse flow direction from direct mud-rotary). This type of rig is used for the construction of large-capacity production water wells and is not suited for small, water quality sampling wells because of the use of drilling muds and the large-diameter hole which is created. A few special reverse-circulation rotary rigs are made with double-wall drill pipe. The drilling water or air is circulated down the annulus between the drill pipes and up inside the inner pipe.

Advantages of the latter method include:

- The formation water is not contaminated by the drilling water.
- Formation samples can be obtained, from known depths.
- When drilling with air, immediate information is available regarding the water-bearing properties of formations penetrated.
- Collapsing of the hole in unconsolidated formations is not as great a problem as when drilling with the normal air-rotary rig.

Disadvantages include:

- Double-wall, reverse-circulation drill rigs are rare and expensive to operate.
- Placing cement grout around the outside of the well casing above a well screen often is difficult, especially when the screen and casing are placed down through the inner drill pipe before the drill pipe is pulled out.

5.2.6 Drill-through Casing Driver

The driven-casing method consists of alternately driving casing (fitted with a sharp, hardened casing shoe) into the ground using a hammer lifted and dropped by the drill rig (or an air-hammer) and cleaning out the casing using a rotary chopping bit and air or water to flush out the materials. The casing is driven down in stages (usually 5 feet per stage); a continuous record is kept of the blows per foot in driving the casing (see SOP GH-1.5). The casing is normally advanced by a 300-pound hammer falling freely through a height of 30 inches. Simultaneous washing and driving of the casing is not recommended. If this procedure is used, the elevations within which wash water is used and in which the casing is driven must be clearly recorded.

The driven casing method is used in unconsolidated formations only. When the boring is to be used for later well installation, the driven casing used should be at least 4 inches larger in diameter than the well casing to be installed. Advantages to this method of drilling include:

- Split-barrel (split-spoon) sampling can be conducted while drilling.
- Well installation is easily accomplished.
- Drill rigs used are relatively small and mobile.
- The use of casing minimizes flow into the hole from upper water-bearing layers; therefore, multiple aquifers can be penetrated and sampled for rough field determinations of some water quality parameters.

Some of the disadvantages include:

- This method can only be used in unconsolidated formations.
- The method is slower than other methods (average drilling progress is 30 to 50 feet per day).
- Maximum depth of the borehole varies with the size of the drill rig and casing diameter used, and the nature of the formations drilled.
- The cost per hour or per foot of drilling may be substantially higher than other drilling methods.
- It is difficult and time consuming to pull back the casing if it has been driven very deep (deeper than 50 feet in many formations).

5.2.7 Cable Tool Drilling

A cable tool rig uses a heavy, solid-steel, chisel-type drill bit ("tool") suspended on a steel cable, which when raised and dropped, chisels or pounds a hole through the soils and rock. Drilling progress may be

expedited by the use of "slip-jars" which serve as a cable-activated down hole percussion device to hammer the bit ahead.

When drilling through the unsaturated zone, some water must be added to the hole. The cuttings are suspended in the water and then bailed out periodically. Below the water table, after sufficient ground water enters the borehole to replace the water removed by bailing, no further water needs to be added. When soft caving formations are encountered, it is usually necessary to drive casing as the hole is advanced to prevent collapse of the hole. Often the drilling can be only a few feet below the bottom of the casing. Because the drill bit is lowered through the casing, the hole created by the bit is smaller than the casing. Therefore, the casing (with a sharp, hardened casing shoe on the bottom) must be driven into the hole (see Section 5.2.5 of this guideline).

Advantages of the cable-tool method include the following:

- Information regarding water-bearing zones is readily available during the drilling. Even relative permeabilities and rough water quality data from different zones penetrated can be obtained by skilled operators.
- The cable-tool rig can operate satisfactorily in all formations, but is best suited for caving, boulder, cobble or coarse gravel type formations (e.g., glacial till) or formations with large cavities above the water table (such as limestones).
- When casing is used, the casing seals formation water out of the hole, preventing down hole contamination and allowing sampling of deeper aquifers for field-measurable water quality parameters.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be collected through the casing.

Disadvantages include:

- Drilling is slow compared with rotary rigs.
- The necessity of driving the casing in unconsolidated formations requires that the casing be pulled back if exposure of selected water-bearing zones is desired. This process complicates the well completion process and often increases costs. There is also a chance that the casing may become stuck in the hole.
- The relatively large diameters required (minimum of 4-inch casing) plus the cost of steel casing result in higher costs compared to rotary drilling methods where casing is not required (e.g., such use of a hollow-stem auger).
- Cable-tool rigs have largely been replaced by rotary rigs. In some parts of the U.S., availability may be difficult.

5.2.8 Jet Drilling (Washing)

Jet drilling, which should be used only for piezometer or vadose zone sampler installation, consists of pumping water or drilling mud down through a small diameter (1/2- to 2-inch) standard pipe (steel or PVC). The pipe may be fitted with a chisel bit or a special jetting screen. Formation materials dislodged by the bit and jetting action of the water are brought to the surface through the annulus around the pipe. As the pipe is jetted deeper, additional lengths of pipe may be added at the surface.

Jet percussion is a variation of the jetting method, in which the casing is driven with a drive weight. Normally, this method is used to place 2-inch-diameter casing in shallow, unconsolidated sand formations, but this method has also been used to install 3- to 4-inch-diameter casings to a depth of 200 feet.

Jetting is acceptable in very soft formations, usually for shallow sampling, and when introduction of drilling water to the formation is acceptable. Such conditions would occur during rough stratigraphic investigation or installation of piezometers for water level measurement. Advantages of this method include:

- Jetting is fast and inexpensive.
- Because of the small amount of equipment required, jetting can be accomplished in locations where access by a normal drilling rig would be very difficult. For example, it would be possible to jet down a well point in the center of a lagoon at a fraction of the cost of using a drill rig.
- Jetting numerous well points just into a shallow water table is an inexpensive method for determining the water table contours, hence flow direction.

Disadvantages include the following:

- A large amount of foreign water or drilling mud is introduced above and into the formation to be sampled.
- Jetting is usually done in very soft formations which are subject to caving. Because of this caving, it is often not possible to place a grout seal above the screen to assure that water in the well is only from the screened interval.
- The diameter of the casing is usually limited to 2 inches.
- Jetting is only possible in very soft formations that do not contain boulders or coarse gravel, and the depth limitation is shallow (about 30 feet without jet percussion equipment).
- Large quantities of water are often needed.

5.2.9 Drilling with a Hand Auger

This method is applicable wherever the formation, total depth of sampling, and the site and groundwater conditions are such as to allow hand auger drilling. Hand augering can also be considered at locations where drill rig access is not possible. All hand auger borings will be performed according to ASTM D1452-80.

Samples should be taken continuously unless otherwise specified by the project plan documents. Any required sampling is performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool. Typical equipment used for sampling and advancing shallow "hand auger" holes are Iwan samplers (which are rotated) or post hole diggers (which are operated like tongs). These techniques are slow but effective where larger pieces of equipment do not have access, and where very shallow holes are desired (less than 15 feet). Surficial soils must be composed of relatively soft and non-cemented formations to allow penetration by the auger.

5.2.10 Rock Drilling and Coring

When soil borings cannot be continued using augers or rotary methods due to the hardness of the soil or when rock or large boulders are encountered, drilling and sampling can be performed using a diamond bit corer in accordance with ASTM D2113.

Drilling is done by rotating and applying downward pressure to the drill rods and drill bit. The drill bit is a circular, hollow, diamond-studded bit attached to the outer core barrel in a double-tube core barrel. The use of single-tube core barrels is not recommended, as the rotation of the barrel erodes the sample and limits its use for detailed geological evaluation. Water or air is circulated down through the drill rods and annular space between the core barrel tubes to cool the bit and remove the cuttings. The bit cuts a core out of the rock which rises into an inner barrel mounted inside the outer barrel. The inner core barrel and rock core are removed by lowering a wire line with a coupling into the drill rods, latching onto the inner barrel and withdrawing the inner barrel. A less efficient variation of this method utilizes a core barrel that cannot be removed without pulling all of the drill rods. This variation is practical only if less than 50 feet of core is required.

Core borings are made through the casing used for the soil borings. The casing must be driven and sealed into the rock formation to prevent seepage from the overburden into the hole to be cored (see Section 5.3 of this guideline). A double-tube core barrel with a diamond bit and reaming shell or equivalent should be used to recover rock cores of a size specified in the project plans. The most common core barrel diameters are listed in Attachment A.

Soft or decomposed rock should be sampled with a driven split-barrel whenever possible or cored with a Denison or Pitcher sampler.

When coring rock, including shale and claystone, the speed of the drill and the drilling pressure, amount and pressure of water, and length of run can be varied to give the maximum recovery from the rock being drilled. Should any rock formation be so soft or broken that the pieces continually fall into the hole causing unsatisfactory coring, the hole should be reamed and a flush-joint casing installed to a point below the broken formation. The size of the flush-joint casing must permit securing the core size specified. When soft or broken rock is anticipated, the length of core runs should be reduced to less than 5 feet to avoid core loss and minimize core disturbance.

Advantages of core drilling include:

- Undisturbed rock cores can be recovered for examination and/or testing.
- In formations in which the cored hole will remain open without casing, water from the rock fractures may be recovered from the well without the installation of a well screen and gravel pack.
- Formation logging is extremely accurate.
- Drill rigs are relatively small and mobile.

Disadvantages include:

- Water or air is needed for drilling.
- Coring is slower than rotary drilling (and more expensive).
- Depth to water cannot accurately be determined if water is used for drilling.
- The size of the borehole is limited.

This drilling method is useful if accurate determinations of rock lithology are desired or if open wells are to be installed into bedrock. To install larger diameter wells in coreholes, the hole must be reamed out to the proper size after boring, using air or mud rotary drilling methods.

5.2.11 Drilling & Support Vehicles

In addition to the drilling method required to accomplish the objectives of the field program, the type of vehicle carrying the drill rig and/or support equipment and its suitability for the site terrain, will often be an additional deciding factor in planning the drilling program. The types of vehicles available are extensive, and depend upon the particular drilling subcontractor's fleet. Most large drilling subcontractors will have a wide variety of vehicle and drill types suited for most drilling assignments in their particular region, while smaller drilling subcontractors will usually have a fleet of much more limited diversity. The weight, size, and means of locomotion (tires, tracks, etc.) of the drill rig must be selected to be compatible with the site terrain to assure adequate mobility between borehole locations. Such considerations also apply to necessary support vehicles used to transport water and/or drilling materials to the drill rigs at the borehole locations. When the drill rigs or support vehicles do not have adequate mobility to easily traverse the site, provisions must be made for assisting equipment, such as bulldozers, winches, timber planking, etc., to maintain adequate progress during the drilling program.

Some of the typical vehicles which are usually available for drill rigs and support equipment are:

- Totally portable drilling/sampling equipment, where all necessary components (tripods, samplers, hammers, catheads, etc.) may be hand carried to the borehole site. Drilling/sampling methods used with such equipment include:

  - Hand augers and lightweight motorized augers.
  - Retractable plug samplers--driven by hand (hammer).
  - Motorized cathead - a lightweight aluminum tripod with a small gas-engine cathead mounted on one leg, used to install small-diameter cased borings. This rig is sometimes called a "monkey on a stick."

- Skid-mounted drilling equipment containing a rotary drill or engine-driven cathead (to lift hammers and drill string), a pump, and a dismounted tripod. The skid is pushed, dragged, or winched (using the cathead drum) between boring locations.

- Small truck-mounted drilling equipment using a Jeep, stake body or other light truck (4 to 6 wheels), upon which are mounted the drill and/or a cathead, a pump, and a tripod or small drilling derrick. On some rigs, the drill and/or a cathead are driven by a power take-off from the truck, instead of by a separate engine.

- Track-mounted drilling equipment is similar to truck-mounted rigs, except that the vehicle used has wide bulldozer tracks for traversing soft ground. Sometimes a continuous-track "all terrain vehicle" is also modified for this purpose. Some types of tracked drill rigs are called "bombardier" or "weasel" rigs.

- Heavy truck-mounted drilling equipment is mounted on tandem or dual tandem trucks to transport the drill, derrick, winches, and pumps or compressors. The drill may be provided with a separate engine or may use a power take-off from the truck engine. Large augers, hydraulic rotary and reverse circulation rotary drilling equipment are usually mounted on such heavy duty trucks. For soft-ground sites, the drilling equipment is sometimes mounted on vehicles having low pressure, very wide diameter tires and capable of floating; these vehicles are called "swamp buggy" rigs.

- Marine drilling equipment is mounted on various floating equipment for drilling borings in lakes, estuaries and other bodies of water. The floating equipment varies, and is often manufactured or customized by the drilling subcontractor to suit specific drilling requirements. Typically, the range of flotation vehicles include:

  - Barrel-float rigs - a drill rig mounted on a timber platform buoyed by empty 55-gallon drums or similar flotation units.

  - Barge-mounted drill rigs.

- Jack-up platforms - drilling equipment mounted on a floating platform having retractable legs to support the unit on the sea or lake bed when the platform is jacked up out of the water.

- Drill ships - for deep ocean drilling.

In addition to the mobility for the drilling equipment, similar consideration must be given for equipment to support the drilling operations. Such vehicles or floating equipment are needed to transport drill water, drilling supplies and equipment, samples, drilling personnel, etc. to and/or from various boring locations.

5.2.12 Equipment Sizes

In planning subsurface exploration programs, care must be taken in specifying the various drilling components, so that they will fit properly in the boring or well.

For drilling open boreholes using rotary drilling equipment, tri-cone drill bits are employed with air, water or drilling mud to remove cuttings and cool the bit. Tri-cone bits are slightly smaller than the holes they drill (i.e., 5-7/8-inch or 7-7/8-inch bits will nominally drill 6-inch and 8-inch holes, respectively).

For obtaining split-barrel samples of a formation, samplers are commonly manufactured in sizes ranging from 2 inches to 3-1/2 inches in outside diameter. However, the most commonly used size is the 2-inch O.D., 1-3/8-inch I.D. split-barrel sampler. When this sampler is used and driven by a 140-pound (± 2-pound) hammer dropping 30 inches (± 1 inch), the procedure is called a Standard Penetration Test, and the blows per foot required to advance the sampler into the formation can be correlated to the formation's density or strength.

In planning the drilling of boreholes using hollow-stem augers or casing, in which thin-wall tube samples or diamond core drilling will be performed, refer to the various sizes and clearances provided in Attachment A of this guideline. Sizes selected must be stated in the project plan documents.

5.2.13 Estimated Drilling Progress

To estimate the anticipated rates of drilling progress for a site, the following must be considered:

- The speed of the drilling method employed.

- Applicable site conditions (e.g., terrain, mobility between borings, difficult drilling conditions in bouldery soils, rubble fill or broken rock, etc.).

- Project-imposed restrictions (e.g., drilling while wearing personal protective equipment, decontamination of drilling equipment, etc.).

Based on recent experience in drilling average soil conditions (no boulders) and taking samples at 5-foot intervals, for moderate depth (30 feet to 50 feet) boreholes (not including installation or development of wells), the following daily rates of total drilling progress may be anticipated for the following drilling methods:

Drilling Method	Average Daily Progress (linear feet)
Hollow-stem augers	75'
Solid-stem augers	50'
Mud-Rotary Drilling	100' (cuttings samples)
Rotosonic Drilling	100'-160' (continuous core)
Reverse-Circulation Rotary	100' (cuttings samples)
Skid-Rig with driven casing	30'
Rotary with driven casing	50'
Cable Tool	30'
Hand Auger	Varies
Continuous Rock Coring	50'

5.3 Prevention of Cross-Contamination

A telescoping or multiple casing technique minimizes the potential for the migration of contaminated groundwater to lower strata below a confining layer. The telescoping technique consists of drilling to a confining layer utilizing a spun casing method with a diamond cutting or augering shoe (a method similar to the rock coring method described in Section 5.2.10, except that larger casing is used) or by using a driven-casing method (see Section 5.2.6 of this guideline) and installing a specified diameter steel well casing. The operation consists of three separate steps. Initially, a drilling casing (usually of 8-inch diameter) is installed followed by installation of the well casing (6-inch-diameter is common for 2-inch wells). This well casing is driven into the confining layer to ensure a tight seal at the bottom of the hole. The well casing is sealed at the bottom with a bentonite-cement slurry. The remaining depth of the boring is drilled utilizing a narrower diameter spun or driven casing technique within the outer well casing. A smaller diameter well casing with an appropriate length of slotted screen on the lower end, is installed to the surface.

Clean sand is placed in the annulus around and to a point of about 2 feet above the screen prior to withdrawal of the drilling casing. The annular space above the screen and to a point 2 feet above the bottom of the outer well casing is sealed with a tremied cement-bentonite slurry which is pressure-grouted or displacement-grouted into the hole. The remaining casing annulus is backfilled with clean material and grouted at the surface, or it is grouted all the way to the surface.

5.4 Cleanout of Casing Prior to Sampling

The boring hole must be completely cleaned of disturbed soil, segregated coarse material and clay adhering to the inside walls of the casing. The cleaning must extend to the bottom edge of the casing and, if possible, a short distance further (1 or 2 inches) to bypass disturbed soil resulting from the advancement of the casing. Loss of wash water during cleaning should be recorded.

For disturbed samples both above and below the water table and where introduction of relatively large volumes of wash water is permissible, the cleaning operation is usually performed by washing the material out of the casing with water; however, the cleaning should never be accomplished with a strong, downward-directed jet which will disturb the underlying soil. When clean out has reached the bottom of the casing or slightly below (as specified above), the string of tools should be lifted one foot off the bottom with the water still flowing, until the wash water coming out of the casing is clear of granular soil particles. In formations where the cuttings contain gravel and other larger particles, it is often useful to repeatedly raise and lower the drill rods and wash bit while washing out the hole, to surge these large particles upward out of the hole. As a time saver, the drilling contractor may be permitted to use a split-barrel (split-spoon) sampler with the ball check valve removed as the clean-out tool, provided the material below the spoon is not disturbed and the shoe of the spoon is not damaged. However, because the ball check valve has been removed, in some formations it may be necessary to install a flap valve or spring sample retainer in the split-spoon bit, to prevent the sample from falling out as the sampler is withdrawn from the hole. The use of jet-type chopping bits is discouraged except where large boulders and cobbles or hard-cemented soils are encountered. If water markedly softens the soils above the water table, clean out should be performed dry with an auger.

For undisturbed samples below the water table, or where wash water must be minimized, clean out is usually accomplished with an appropriate diameter clean out auger. This auger has cutting blades at the bottom to carry loose material up into the auger, and up-turned water jets just above the cutting blades to carry the removed soil to the surface. In this manner, there is a minimum of disturbance at the top of the material to be sampled. If any gravel material washes down into the casing and cannot be removed by the clean out auger, a split-barrel sample can be taken to remove it; bailers and sandpumps should not be used. For undisturbed samples above the groundwater table, all operations must be performed in a dry manner.

If all of the cuttings created by drilling through the overlying formations are not cleaned from the borehole prior to sampling, some of the problems which may be encountered during sampling include:

- When sampling is attempted through the cuttings remaining in the borehole, all or part of the sampler may become filled with the cuttings. This limits the amount of sample from the underlying formation which can enter and be retained in the sampler, and also raises questions as to the validity of the sample.

- If the cuttings remaining in the borehole contain coarse gravel and/or other large particles, these may block the bit of the sampler and prevent any materials from the underlying formation from entering the sampler when the sampler is advanced.

- In cased borings, should sampling be attempted through cuttings which remain in the lower portion of the casing, these cuttings could cause the sampler to become bound into the casing, such that it becomes very difficult to either advance or retract the sampler.

- When sampler blow counts are used to estimate the density or strength of the formation being sampled, the presence of cuttings in the borehole will usually give erroneously high sample blow counts.

To confirm that all cuttings have been removed from the borehole prior to attempting sampling, it is important that the site geologist measure the "stickup" of the drill string. This is accomplished by measuring the assembled length of all drill rods and bits or samplers (the drill string) as they are lowered to the bottom of the hole, below some convenient reference point of the drill string, then measuring the height of this reference point above the ground surface. The difference of these measurements is the

depth of the drill string (lower end of the bit or sampler) below the ground surface, which must then be compared with the depth of sampling required (installed depth of casing or depth of borehole drilled). If the length of drill string below grade is more than the drilled or casing depth, the borehole has been cleaned too deeply, and this deeper depth of sampling must be recorded on the log. If the length of drill string below grade is less than the drilled or casing depth, the difference represents the thickness of cuttings which remain in the borehole. In most cases, an inch or two of cuttings may be left in the borehole with little or no problem. However, if more than a few inches of cuttings are encountered, the borehole must be recleaned prior to attempting sampling.

5.5 Materials of Construction

The effects of monitoring well construction materials on specific chemical analytical parameters are described and/or referenced in SOP GH-2.8. However, there are several materials used during drilling, particularly drilling fluids and lubricants, which must be used with care to avoid compromising the representativeness of soil and ground water samples.

The use of synthetic or organic polymer slurries is not permitted at any location where soil samples for chemical analysis are to be collected. These slurry materials could be used for installation of long-term monitoring wells, but the early time data in time series collection of ground water data may then be suspect. If synthetic or organic polymer muds are proposed for use at a given site, a complete written justification including methods and procedures for their use must be provided by the site geologist and approved by the Project Manager. The specific slurry composition and the concentration of suspected contaminants for each site must be known.

For many drilling operations, potable water is an adequate lubricant for drill stem and drilling tool connections. However, there are instances, such as drilling in tight clayey formations or in loose gravels, when threaded couplings must be lubricated to avoid binding. In these instances, to be determined in the field by the judgment of the site geologist and noted in the site logbook, and only after approval by the Project Manager, a vegetable oil or silicone-based lubricant should be used. Petroleum based greases, etc. will not be permitted. Samples of lubricants used must be provided and analyzed for chemical parameters appropriate to the given site.

5.6 Subsurface Soil Samples

Subsurface soil samples are used to characterize subsurface stratigraphy. This characterization can indicate the potential for migration of chemical contaminants in the subsurface. In addition, definition of the actual migration of contaminants can be obtained through chemical analysis of the soil samples. Where the remedial activities may include in-situ treatment or excavation and removal of the contaminated soil, the depth and areal extent of contamination must be known as accurately as possible.

Engineering and physical properties of soil may also be of interest should site construction activities be planned. Soil types, grain size distribution, shear strength, compressibility, permeability, plasticity, unit weight, and moisture content are some of the physical characteristics that may be determined for soil samples.

Penetration tests are also described in this procedure. The tests can be used to estimate various physical and engineering parameters such as relative density, unconfined compressive strength, and consolidation characteristics of soils.

Surface protocols for various soil sampling techniques are discussed in SOP SA-1.3. Continuous-core soil sampling and rock coring are discussed below. The procedures described here are representative of

a larger number of possible drilling and sampling techniques. The choice of techniques is based on a large number of variables such as cost, local geology, etc. The final choice of methods must be made with the assistance of drilling subcontractors familiar with the local geologic conditions. Alternative techniques must be based upon the underlying principles of quality assurance implicit in the following procedures.

The CME continuous sample tube system provides a method of sampling soil continuously during hollow-stem augering. The 5-foot sample barrel fits within the lead auger of a hollow-auger column. The sampling system can be used with a wide range of I.D. hollow-stem augers (from 3-1/4-inch to 8-1/4-inch I.D.). This method has been used to sample many different materials such as glacial drift, hard clays and shales, mine tailings, etc. This method is particularly used when SPT samples are not required and a large volume of material is needed. Also, this method is useful when a visual description of the subsurface lithology is required. Rotosonic drilling methods also provide a continuous soil sample.

5.7 Rock Sampling (Coring) (ASTM D2113-83)

Rock coring enables a detailed assessment of borehole conditions to be made, showing precisely all lithologic changes and characteristics. Because coring is an expensive drilling method, it is commonly used for shallow studies of 500 feet or less, or for specific intervals in the drill hole that require detailed logging and/or analyzing. Rock coring can, however, proceed for thousands of feet continuously, depending on the size of the drill rig, and yields better quality data than air-rotary drilling, although at a substantially reduced drilling rate. Rate of drilling varies widely, depending on the characteristics of lithologies encountered, drilling methods, depth of drilling, and condition of drilling equipment. Average output in a 10-hour day ranges from 40 to over 200 feet. Down hole geophysical logging or television camera monitoring is sometimes used to complement the data generated by coring.

Borehole diameter can be drilled to various sizes, depending on the information needed. Standard sizes of core barrels (showing core diameter) and casing are shown in Figure 1.

Core drilling is used when formations are too hard to be sampled by soil sampling methods and a continuous solid sample is desired. Usually, soil samples are used for overburden, and coring begins in sound bedrock. Casing is set into bedrock before coring begins to prevent loose material from entering the borehole, to prevent loss of drilling fluid, and to prevent cross-contamination of aquifers.

Drilling through bedrock is initiated by using a diamond-tipped core bit threaded to a drill rod (outer core barrel) with a rate of drilling determined by the downward pressure, rotation speed of drill rods, drilling fluid pressure in the borehole, and the characteristics of the rock (mineralogy, cementation, weathering).

FIGURE 1

STANDARD SIZES OF CORE BARRELS AND CASING

Coring Bit Size	Nominal*		Set Size*	
	O.D.	I.D.	O.D.	I.D.
RWT	1 5/32	3/4	1.160	0.735
EWT	1 1/2	29/32	1.470	0.905
EX, EXL, EWG, EWM	1 1/2	13/16	1.470	0.845
AWT	1 7/8	1 9/32	1.875	1.281
AX, AXL, AWG, AWM	1 7/8	1 3/16	1.875	1.185
BWT	2 3/8	1 3/4	2.345	1.750
BX, BXL, BWG, BWM	2 3/8	1 5/8	2.345	1.655
NWT	3	2 5/16	2.965	2.313
NX, NXL, NWG, NWM	3	2 1/8	2.965	2.155
HWT	3 29/32	3 3/16	3.889	3.187
HWG	3 29/32	3	3.889	3.000
2 3/4 x 3 7/8	3 7/8	2 3/4	3.840	2.690
4 x 5 1/2	5 1/2	4	5.435	3.970
6 x 7 3/4	7 3/4	6	7.655	5.970
AX Wire line ___|___/	1 7/8	1	1.875	1.000
BX Wire line ___|___/	2 3/8	1 7/16	2.345	1.437
NX Wire line ___|___/	3	1 15/16	2.965	1.937

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

___|___/ Wire line dimensions and designations may vary according to manufacturer.

FIGURE 1
STANDARD SIZES OF CORE BARRELS AND CASING
PAGE TWO

Size Designations		Casing O.D., Inches	Casing Coupling		Casing bit O.D., Inches	Core barrel bit O.D., Inches*	Drill rod O.D., Inches	Approximate Core Diameter	
Casing; Casing coupling; Casing bits; Core barrel bits	Rod; rod couplings		O.D., Inches	I.D., Inches				Normal, Inches	Thinwall, Inches
RX	RW	1.437	1.437	1.188	1.485	1.160	1.094	---	0.735
EX	E	1.812	1.812	1.500	1.875	1.470	1.313	0.845	0.905
AX	A	2.250	2.250	1.906	2.345	1.875	1.625	1.185	1.281
BX	B	2.875	2.875	2.375	2.965	2.345	1.906	1.655	1.750
NX	N	3.500	3.500	3.000	3.615	2.965	2.375	2.155	2.313
HX	HW	4.500	4.500	3.938	4.625	3.890	3.500	3.000	3.187
RW	RW	1.437	Flush Joint	No Coupling	1.485	1.160	1.094	---	0.735
EW	EW	1.812			1.875	1.470	1.375	0.845	0.905
AW	AW	2.250			2.345	1.875	1.750	1.185	1.281
BW	BW	2.875			2.965	2.345	2.125	1.655	1.750
NW	NW	3.500			3.615	2.965	2.625	2.155	2.313
HW	HW	4.500			4.625	3.890	3.500	3.000	3.187
PW	---	5.500			5.650	---	---	---	---
SW	---	6.625			6.790	---	---	---	---
UW	---	7.625			7.800	---	---	---	---
ZW	---	8.625			8.810	---	---	---	---
---	AX __|__\	---	---	---	---	1.875	1.750	1.000	---
---	BX __|__\	---	---	---	---	2.345	2.250	1.437	---
---	NX __|__\	---	---	---	---	2.965	2.813	1.937	---

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

___|___/ Wire line dimensions and designations may vary according to manufacturer.

NOMINAL DIMENSIONS FOR DRILL CASINGS AND ACCESSORIES. (DIAMOND CORE DRILL MANUFACTURERS ASSOCIATION). 288-D-2889

5.7.1 Diamond Core Drilling

A penetration of typically less than 6 inches per 50 blows using a 140-lb. hammer dropping 30 inches with a 2-inch split-barrel sampler shall be considered an indication that soil sampling methods may not be applicable and that coring may be necessary to obtain samples.

When formations are encountered that are too hard to be sampled by soil sampling methods, the following diamond core drilling procedure may be used:

- Firmly seat a casing into the bedrock or the hard material to prevent loose materials from entering the hole and to prevent the loss of drilling fluid return. Level the surface of the rock or hard material when necessary by the use of a fishtail or other bits. If the drill hole can be retained open without the casing and if cross-contamination of aquifers in the unconsolidated materials is unlikely, leveling may be omitted.

- Begin the core drilling using a double-tube swivel-core barrel of the desired size. After drilling no more than 10 feet (3 m), remove the core barrel from the hole and take out the core. If the core blocks the flow of the drilling fluid during drilling, remove the core barrel immediately. In soft materials, a large starting size may be specified for the coring tools; where local experience indicates satisfactory core recovery or where hard, sound materials are anticipated, a smaller size or the single-tube type may be specified and longer runs may be drilled. NX/NW size coring equipment is the most commonly used size.

- When soft materials are encountered that produce less than 50 percent recovery, stop the core drilling. If soil samples are desired, secure such samples in accordance with the procedures described in ASTM Method D 1586 (Split-barrel Sampling) or in Method D 1587 (Thin-Walled Tube Sampling); sample soils per SOP SA-1.3. Resume diamond core drilling when refusal materials are again encountered.

- Since rock structures and the occurrence of seams, fissures, cavities, and broken areas are among the most important items to be detected and described, take special care to obtain and record these features. If such broken zones or cavities prevent further advance of the boring, one of the following three steps shall be taken: (1) cement the hole; (2) ream and case; or (3) case and advance with the next smaller size core barrel, as conditions warrant.

- In soft, seamy, or otherwise unsound rock, where core recovery may be difficult, M-design core barrels may be used. In hard, sound rock where a high percentage of core recovery is anticipated, the single-tube core barrel may be employed.

5.7.2 Rock Sample Preparation and Documentation

Once the rock coring has been completed and the core recovered, the rock core shall be carefully removed from the barrel, placed in a core tray (previously labeled "top" and "bottom" to avoid confusion), classified, and measured for percentage of recovery as well as the rock quality designation (RQD). Each core shall be described, classified, and logged using a uniform system as presented in SOP GH-1.5. If moisture content will be determined or if it is desirable to prevent drying (e.g., to prevent shrinkage of clay formations) or oxidation of the core, the core shall be wrapped in plastic sleeves immediately after logging. Each plastic sleeve shall be labeled with indelible ink. The boring number, run number, and the footage represented in each sleeve shall be included, as well as designating the top and bottom of the core run.

After sampling, rock cores shall be placed in the sequence of recovery in well-constructed wooden boxes provided by the drilling contractor. Rock cores from two different borings shall not be placed in the same core box unless accepted by the Project Geologist. The core boxes shall be constructed to accommodate at least 20 linear feet of core in rows of approximately 5 feet each and shall be constructed with hinged tops secured with screws, and a latch (usually a hook and eye) to keep the top securely fastened down. Wood partitions shall be placed at the end of each core run and between rows.

The depth from the surface of the boring to the top and bottom of the drill run and run number shall be marked on the wooden partitions with indelible ink. A wooden partition (wooden block) shall be placed at the end of each run with the depth of the bottom of the run written on the block. These blocks will serve to separate successive core runs and indicate depth intervals for each run. The order of placing cores shall be the same in all core boxes. Rock core shall be placed in the box so that, when the box is open, with the inside of the lid facing the observer, the top of the cored interval contained within the box is in the upper left corner of the box, and the bottom of the cored interval is in the lower right corner of the box. The top and bottom of each core obtained and its true depth shall be clearly and permanently marked on each box. The width of each row must be compatible with the core diameter to prevent lateral movement of the core in the box. Similarly, an empty space in a row shall be filled with an appropriate filler material or spacers to prevent longitudinal movement of the core in the box.

The inside and outside of the core-box lid shall be marked by indelible ink to show all pertinent data on the box's contents. At a minimum, the following information shall be included:

- Project name.
- Project number.
- Boring number.
- Run numbers.
- Footage (depths).
- Recovery.
- RQD (%).
- Box number and total number of boxes for that boring (Example: Box 5 of 7).

For easy retrieval when core boxes are stacked, the sides and ends of the box shall also be labeled and include project number, boring number, top and bottom depths of core and box number.

Prior to final closing of the core box, a photograph of the recovered core and the labeling on the inside cover shall be taken. If moisture content is not critical, the core shall be wetted and wiped clean for the photograph. (This will help to show true colors and bedding features in the cores).

6.0 REFERENCES

Acker Drill Co., 1958. Basic Procedures of Soil Sampling. Acker Drill Co., Scranton, Pennsylvania.

American Institute of Steel Construction, 1978. Manual of Steel Construction, 7th Edition. American Institute of Steel Construction, New York, New York.

American Society for Testing and Materials, 1987. ASTM Standards D1587-83, D1586-84, and D1452-80. ASTM Annual Book of Standards, ASTM, Philadelphia, Pennsylvania, Vol. 4.08.

American Society for Testing and Materials, 1989. Standard Practice for Diamond Core Drilling for Site Investigation. ASTM Method D2113-83 (reapproved 1987), Annual Book of Standards, ASTM, Philadelphia, Pennsylvania.

Barcelona, M. J., J. P. Gibb and R. A. Miller, 1983. A Guide to the Selection of Material for Monitoring Well Construction and Ground Water Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

BOART Longyear Co., Sonic Drilling. Environmental Drilling Division, Andova, Minnesota.

Central Mine Equipment Company, Drilling Equipment, St. Louis, Missouri.

Dept. of the Navy, Naval Facilities Engineering Command, 1982. Soil Mechanics Design Manual 7.1.

Driscoll, Fletcher G., 1986. Groundwater and Wells, 2nd Edition. Johnson Division, St. Paul, Minnesota.

Procedure GH-1.5 - Borehole and Sample Logging.

Scalf, M. R., J. F. McNabb, W. J. Dunlap, R. L. Crosby and J. Fryberger, 1981. Manual of Ground-Water Sampling Procedures. NWWA/EPA Series. Kerr Environmental Research Laboratory, Office of Research and Development, U.S. EPA, Ada, Oklahoma.

U.S. Department of the Interior, 1974, Earth Manual, A Water Resources Technical Publication, 810 pages.

U.S. EPA, 1980. Procedure Manual for Ground Water Monitoring at Solid Waste Disposal Facilities. SW-611. Office of Solid Waste, U.S. EPA, Cincinnati, Ohio.

W. L. Acker III, 1974. Basic Procedures for Soil Sampling and Core Drilling. Acker Drill Co., Inc., Scranton, Pennsylvania.

ATTACHMENT A

DRILLING EQUIPMENT SIZES

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Hollow-stem augers (Ref. 7)	6 1/4	5	2 1/4	
	6 3/4	5 3/4	2 3/4	---
	7 1/4	6 1/4	3 1/4	---
	13 1/4	12	6	---
Thin Wall Tube Samplers (Ref. 7)	---	2	1 7/8	---
	---	2 1/2	2 3/8	---
	---	3	2 7/8	---
	---	3 1/2	3 3/8	---
	---	4 1/2	4 3/8	---
	---	5	4 3/4	---
Drill Rods (Ref. 7)	RW	1 3/32	23/32	13/32
	EW	1 3/8	15/16	7/16
	AW	1 3/4	1 1/4	5/8
	BW	2 1/8	1 3/4	3/4
	NW	2 5/8	2 1/4	1 3/8
	HW	3 1/2	3 1/16	2 3/8
	E	1 5/16	7/8	7/16
	A	1 5/8	1 1/8	9/16
	B	1 7/8	1 1/4	5/8
	N	2 3/8	2	1
				Wall Thickness (Inches)
Driven External Coupled Extra Strong Steel* Casing (Ref. 8)	2 1/2	2.875	2.323	0.276
	3	3.5	2.9	0.300
	3 1/2	4.0	3.364	0.318
	4	4.5	3.826	0.337
	5	5.63	4.813	0.375
	6	6.625	5.761	0.432
	8	8.625	7.625	0.500
	10	10.750	9.750	0.500
	12	12.750	11.750	0.500

* Add twice the casing wall thickness to casing O.D. to obtain the approximate O.D. of the external pipe couplings.

ATTACHMENT A
DRILLING EQUIPMENT SIZES
PAGE TWO

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Flush Coupled Casing (Ref. 7)	RX	1 7/16	1 3/16	1 3/16
	EX	1 13/16	1 5/8	1 1/2
	AX	2 1/4	2	1 29/32
	BX	2 7/8	2 9/16	2 3/8
	NX	3 1/2	3 3/16	3
	HX	4 1/2	4 1/8	3 15/16
Flush Joint Casing (Ref. 7)	RW	1 7/16	1 3/16	
	EW	1 13/16	1 1/2	
	AW	2 1/4	1 29/32	
	BW	2 7/8	2 3/8	
	NW	3 1/2	3	
	HW	4 1/2	4	
	PW	5 1/2	5	
	SW	6 5/8	6	
	UW	7 5/8	7	
	ZW	8 5/8	8	
Diamond Core Barrels (Ref. 7)	EWM	1 1/2	7/8**	
	AWM	1 7/8	1 1/8**	
	BWM	2 3/8	1 5/8**	
	NWM	3	2 1/8	
	HWG	3 7/8	3	
	2 3/4 x 3 7/8	3 7/8	2 11/16	
	4 x 5 1/2	5 1/2	3 15/16	
	6 x 7 3/4	7 3/4	5 15/16	
	AQ (wireline)	1 57/64	1 1/16**	
	BQ (wireline)	2 23/64	1 7/16**	
	NQ (wireline)	2 63/64	1 7/8	
	HQ (wireline)	3 25/32	2 1/2	

** Because of the fragile nature of the core and the difficulty to identify rock details, use of small-diameter core (1 3/8") is not recommended.

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number HS-1.0	Page 1 of 15
		Effective Date 12/03	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Health & Safety	
Subject UTILITY LOCATING AND EXCAVATION CLEARANCE		Approved D. Senovich	

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES **3**

5.0 PROCEDURES **3**

5.1 BURIED UTILITIES 3
5.2 OVERHEAD POWER LINES 5

6.0 UNDERGROUND LOCATING TECHNIQUES **5**

6.1 GEOPHYSICAL METHODS 5
6.2 PASSIVE DETECTION SURVEYS 6
6.3 INTRUSIVE DETECTION SURVEYS 6

7.0 INTRUSIVE ACTIVITIES SUMMARY **7**

8.0 REFERENCES **8**

ATTACHMENTS

1 Listing of Underground Utility Clearance Resources 9
2 Frost Line Penetration Depths by Geographic Location 11
3 Utility Clearance Form 12
4 OSHA Letter of Interpretation 13

1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.

2. A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scares and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.

4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.

5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain

6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.

7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.

8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly though conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

Nominal Voltage	Minimum Clearance
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.

2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

   Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.

4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
OSHA 29 CFR 1926(b)(2)
OSHA 29 CFR 1926(b)(3)
TtNUS Utility Locating and Clearance Policy
TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

ONE-CALL SYSTEMS INTERNATIONAL CONDENSED DIRECTORY

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5348

Arkansas
Arkansas One Call System, Inc.
1-800-482-8998

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-922-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8989

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007

Louisiana
Louisiana One Call System, Inc.
1-800-272-3020

Maine
Dig Safe System, Inc.
1-888-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-888-344-7233

Michigan
Miss Dig System, Inc.
1-800-482-7171

Minnesota
Gopher State One Call
1-800-252-1166

Mississippi
Mississippi One-Call System, Inc
1-800-227-6477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344

Nebraska
Diggers Hotline of Nebraska
1-800-331-5666

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-888-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400

New York
Dig Safely New York
1-800-962-7962
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-632-4949

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0988

Oklahoma
Call Okie
1-800-522-6543

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-888-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111

ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah
Blue Stakes of Utah
1-800-662-4111

Vermont
Dig Safe System, Inc.
1-888-344-7233

Virginia
Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington
Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating Council
509-456-8000

West Virginia
Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin
Diggers Hotline, Inc.
1-800-242-8511

Wyoming
Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia
Miss Utility
1-800-257-7777

Alberta
Alberta One-Call Corporation
1-800-242-3447

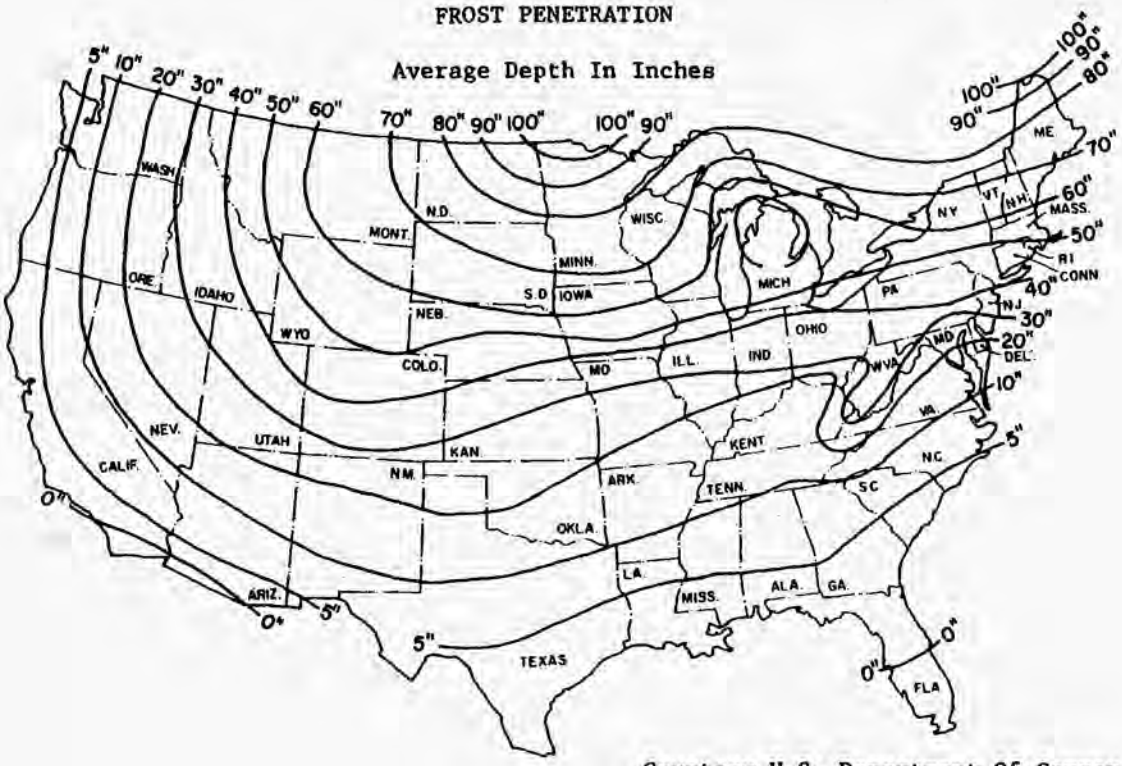
British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-400-2255

Quebec
Info-Excavation
1-800-663-9228

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

ATTACHMENT 3
UTILITY CLEARANCE FORM

Client: _____ Project Name: _____

Project No.: _____ Completed By: _____

Location Name: _____ Work Date: _____

Excavation Method/Overhead Equipment: _____

			Circle One
1.	Underground Utilities		
	a)	Review of existing maps?	yes no N/A
	b)	Interview local personnel?	yes no N/A
	c)	Site visit and inspection?	yes no N/A
	d)	Excavation areas marked in the field?	yes no N/A
	e)	Utilities located in the field?	yes no N/A
	f)	Located utilities marked/added to site maps?	yes no N/A
	g)	Client contact notified Name _____ Telephone: _____ Date: _____	yes no N/A
	g)	State One-Call agency called? Caller: _____ Ticket Number: _____ Date: _____	yes no N/A
	h)	Geophysical survey performed? Survey performed by: _____ Method: _____ Date: _____	yes no N/A
	j)	Hand excavation performed (with concurrent use of utility detection device)? Completed by: _____ Total depth: _____ feet Date: _____	yes no N/A
	j)	Trench/excavation probed? Probing completed by: _____ Depth/frequency: _____ Date: _____	yes no N/A

			Present Absent
2.	Overhead Utilities		
	a)	Determination of nominal voltage	yes no N/A
	b)	Marked on site maps	yes no N/A
	c)	Necessary to lockout/insulate/re-route	yes no N/A
	d)	Document procedures used to lockout/insulate/re-route	yes no N/A
	e)	Minimum acceptable clearance (SOP Section 5.2): _____	

3. Notes:

_____

_____

_____

Approval:

_____ _____

Site Manager/Field Operations Leader Date

c: PM/Project File
Program File

ATTACHMENT 4
OSHA LETTER OF INTERPRETATION

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

Question: *Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651(Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

> When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

ATTACHMENT 4 (Continued)

> When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, "acceptable means" must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either "other acceptable means" or "safe and acceptable means." The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified "careful probing or hand digging" as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language "to allow other, *equally effective means* of locating such installations." The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used *where the utilities have not been marked and detection equipment is not being used* – "probing with hand-held tools." This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

> OSHA received two comments * * * and input from ACCSH [OSHA's Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping 'such as probing with hand-held tools' from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of "acceptable means" in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a "shooter" (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an "acceptable means" for locating underground utilities.

ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA=s interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at http://www.osha.gov.

APPENDIX B

FIELD DOCUMENTATION FORMS

Tetra Tech NUS, Inc.

BORING LOG

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): ____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 4869 | PAGE ____ OF _____

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

DATE / YEAR	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAB (G) COMP (C)	No. OF CONTAINERS	CONTAINER TYPE PLASTIC (P) or GLASS (G) / PRESERVATIVE USED / TYPE OF ANALYSIS	COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R
FORM NO. TtNUS-001

Tetra Tech NUS, Inc.

PROJECT: _____ **JOB #:** _____
LOCATION: _____ **DATE:** _____
PROJECT MANAGER: _____ **FOL:** _____

DAILY ACTIVITIES CHECKLIST

Startup Checklist

Activity	Yes	No	N/A
Pertinent site activities/information entered into site logbook			
All onsite personnel listed in logbook			
Required medical information onsite for all workers (TtNUS and Subcontractors)			
Required MSDS's onsite			
Proper equipment calibrations performed (list equipment)			
1 _____			
2 _____			
3 _____			
4 _____			
Calibration logs filled out			
Tailgate H&S meeting held prior to beginning field activities			
Required work permits filled out/signed			
Required utility clearances obtained			
Required PPE onsite and in use			
Information required to be posted is in place (OSHA poster, hospital route, key phone numbers, etc.)			

Exit Checklist

Activity	Yes	No	N/A
Logbooks completely and comprehensively filled out			
Field forms complete and accounted for/properly filed			
Samples properly packaged/shipped			
COCs faxed to appropriate in-house personnel			
All equipment accounted for, on charge if needed, and properly secured			
All personnel accounted for			
Arrangements made for upcoming work (permits, clearances, equipment, etc.)			
Site properly secured			

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

TETRA TECH NUS, INC.

FIELD INSTRUMENT CALIBRATION LOG

INSTRUMENT NAME: _____ MODEL No.: _____

SERIAL No.: _____ DECAL No.: _____ TETRA TECH NUS CHARGE No. _____

CALIBRATION DATE	INITIAL READING	PROCEDURE	FINAL READING	SIGNATURE	COMMENTS

TtNUS Form 0007

TETRA TECH NUS, INC.

FIELD MODIFICATION RECORD

Site Name: _____ Location: _____

Project Number: _____ Task Assignment: _____

To: _____ Location: _____ Date: _____

Description:_____

_____

_____

_____

Reason for Change: _____

_____

_____

Recommended Action: _____

_____

_____

Field Operations Leader (Signature): _____ Date: _____

Disposition/Action:_____

_____

_____

Project Manager (Signature): _____ Date: _____

Distribution:

Program Manager:	_____	Others as Required:
Project Manager:	_____	_____
Quality Assurance Officer:	_____	_____
Field Operations Leader:	_____	_____
Project File:	_____	_____

TtNUS Form 0003

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____

Location: _____ **Personnel:** _____

Weather Conditions: _____ **Measuring Device:** _____

Tidally Influenced: **Yes** ____ **No** ____ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Total Well Depth (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Groundwater Elevation (feet)*	Comments

* All measurements to the nearest 0.01 foot

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Domestic Well Data
[] Monitoring Well Data
[] Other Well Type: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

Date: / Time:	**Color** (Visual)	**pH** (S.U.)	**S.C.** (mS/cm)	**Temp.** (^{0}C)	**Turbidity** (NTU)	**DO** (mg/l)	**Salinity** (%)	Other
Method:								

PURGE DATA:

Date:	**Volume**	**pH**	**S.C.**	**Temp.**	**Turbidity**	**DO**	**Salinity**	Other
Method:								
Monitor Reading (ppm):								
Well Casing Diameter & Material Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	**Preservative**	**Container Requirements**	**Collected**

OBSERVATIONS / NOTES:

Circle if Applicable:

MS/MSD | **Duplicate ID No.:**

Signature(s):

Tetra Tech NUS, Inc.

MONITORING WELL DEVELOPMENT RECORD

Site: _____ Depth to Bottom (ft.): _____ Project Name: _____
Well: _____ Static Water Level Before (ft.): _____ Project Number: _____
Date Installed: _____ Static Water Level After (ft.): _____ Site Geologist: _____
Date Developed: _____ Screen Length (ft.): _____ Drilling Co.: _____
Dev. Method: _____ Specific Capacity: _____
Pump Type: _____ Casing ID (in.): _____

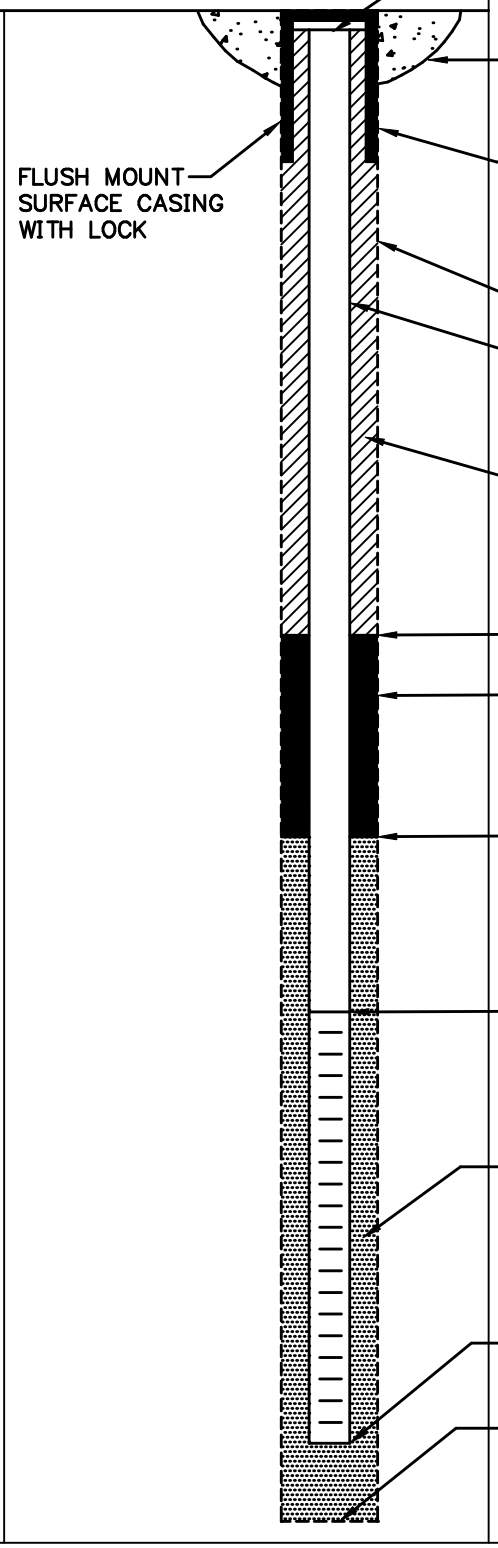
Time	Estimated Sediment Thickness (Ft.)	Cumulative Water Volume (Gal.)	Water Level Readings (Ft. below TOC)	Temperature (Degrees C)	pH	Specific Conductance (Units _____)	Turbidity (NTU)	Remarks (odor, color, etc.)

WELL NO.: _____

Tetra Tech NUS, Inc.

OVERBURDEN
MONITORING WELL SHEET
FLUSH - MOUNT

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION TOP OF RISER: _____

TYPE OF SURFACE SEAL: _____

_____

TYPE OF PROTECTIVE CASING: _____

_____

I.D. OF PROTECTIVE CASING: _____

DIAMETER OF HOLE: _____

TYPE OF RISER PIPE: _____

_____

RISER PIPE I.D.: _____

TYPE OF BACKFILL/SEAL: _____

_____

ELEVATION/DEPTH TOP OF SEAL: _____ / _____

TYPE OF SEAL: _____

_____

ELEVATION/DEPTH TOP OF SAND: _____ / _____

ELEVATION/DEPTH TOP OF SCREEN: _____ / _____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

TYPE OF SAND PACK: _____

_____

DIAMETER OF HOLE IN BEDROCK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: _____ / _____

ELEVATION / DEPTH BOTTOM OF SAND: _____ / _____

ELEVATION/DEPTH BOTTOM OF HOLE: _____ / _____

BACKFILL MATERIAL BELOW SAND: _____

_____

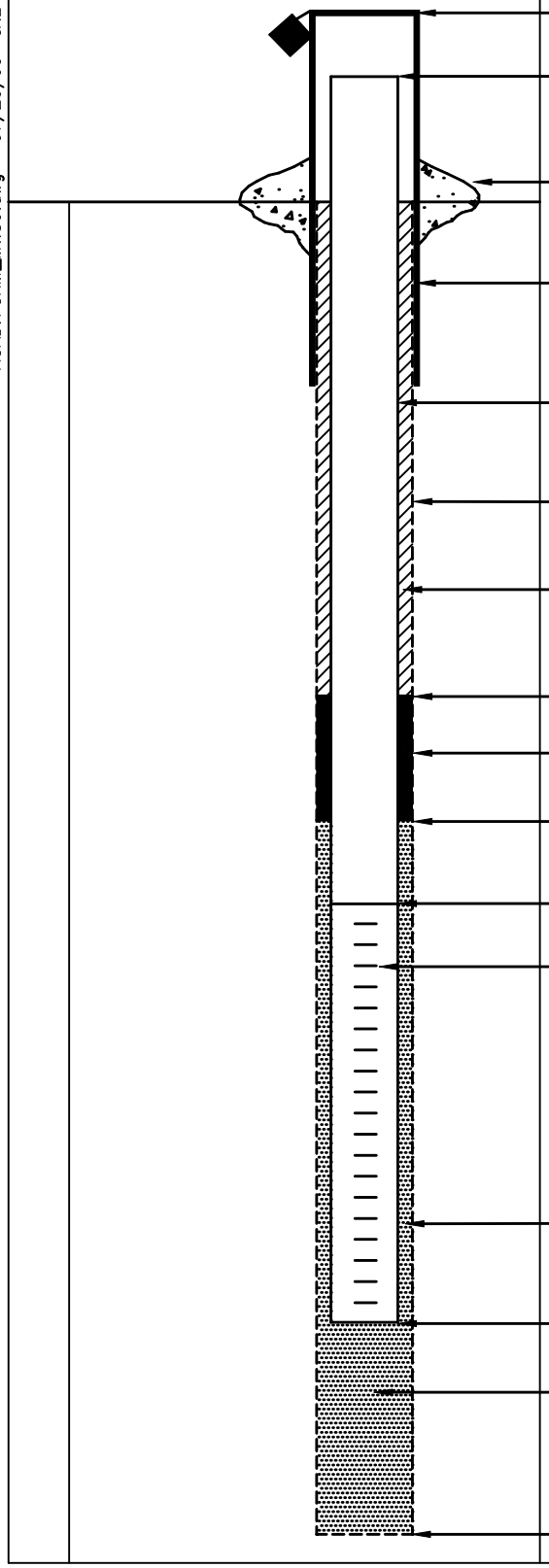
ACAD: FORM_MWFM.dwg 07/20/99 INL

WELL NO.: _____

Tetra Tech NUS, Inc.

OVERBURDEN
MONITORING WELL SHEET
STICK-UP

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION/HEIGHT OF TOP OF SURFACE CASING: ____/____

ELEVATION/HEIGHT OF TOP OF RISER PIPE: ____/____

TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____

TYPE OF SURFACE CASING: _____

RISER PIPE I.D.: _____

TYPE OF RISER PIPE: _____

BOREHOLE DIAMETER: _____

TYPE OF BACKFILL: _____

ELEVATION/DEPTH TOP OF SEAL: ____/____

TYPE OF SEAL: _____

DEPTH TOP OF SAND PACK: _____

ELEVATION/DEPTH TOP OF SCREEN: ____/____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

I.D. OF SCREEN: _____

TYPE OF SAND PACK: _____

ELEVATION/DEPTH BOTTOM OF SCREEN: ____/____

ELEVATION/DEPTH BOTTOM OF SAND PACK: ____/____

BACKFILL MATERIAL BELOW SAND: _____

ELEVATION/DEPTH OF HOLE: ____/____

ACAD:FORM_MWSU.dwg 07/20/99 INL

TETRA TECH NUS, INC.

PHOTOIONIZATION DETECTOR FIELD CALIBRATION LOG

Serial No.: _____ Model No.: _____ Decal No.: _____

Site Name/Location: _____ Tetra Tech NUS Charge No.: _____

CALIBRATION DATE	STANDARD GAS-ISOBUTYLENE	(AM) CALIBRATION READING Isobutylene Equiv. (ppm)	(PM) CALIBRATION CHECK Isobutylene Equiv. (ppm)	SIGNATURE	COMMENTS
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	

TtNUS Form 0006

TETRA TECH NUS, INC.

Quarterly LTM: Round 1 2008

PURGE DATA SHEET - "LOW FLOW" GROUNDWATER

Site Name:
Sample ID:

Tetra Tech NUS Charge No. / CTO Page 1 of
QC: _____ (If applicable)

Sample Method: Low Stress (flow) with Peristaltic Pump
Depth Sampled: _____ ft bgs Screen Int. Depth _____ ft bgs
Sample Date & Time: 4/___ /2008 _____hours _____(Dup Time)
Sampler(s):
Data Recorded By:_____ Signature: _____ ;
Notes: Water Depth MP = top of riser (TOR); NAPL signal at start (Yes/No); if yes, report depths/describe thickness_____; Visual Evidence of Sheen (Yes/No) _____;
Olfactory Evidence of Odor (Yes/No)_____;
Weather: _____

H&S Survey Meter _____PPM Field Instrument Group A/B/C/D
Pre-tubing insertion WL _____ft btor; Post –tubing insertion WL _____ft btor

Field Analysis: Fe2+

Sampling Hierarchy and Lab Analyses:
1. VOC **2.** EDB/DBCP **3.** VPH **4.** Pest/PCB **5a.** Metals(unfiltered)
5b. Metals (filtered) **6.** Cyanide **7.** SVOC/PAH **8.** Herbicides **9.** COD
10. Alk, Nitrate, TDS, Chloride, Sulfate **11.** EPH

(Also see separate sample logsheet for gw)

Clock Time 24hr	Water Depth below MP ft	Pump Dial 1	Purge Rate ml/min	Cum. Volume Purged Gals.	Temp °C	Spec. Cond. 2 uS/cm	pH (S.U.)	ORP/Eh3 mv	DO mg/L	Turbidity NTU	Comments
Acceptance Criteria: <0.3 ft (drawdown)					3%	3%	+/- 1.0 S.U.	+/- 10mV	10%	10%	

TtNUS Form 0009

Saturated Screen Volume (gallons) _____ (2" screen = 0.163 gals/ft of depth; 4" = 0.653 gals/ft; 6" = 1.469gals/ft)

1. Pump dial setting (for example: hertz, cycle/min, etc.)
2. Siemens per cm (same as umhos/cm) at 25 °C.
3. Oxidation reduction potential (stand in for Eh).

TETRA TECH NUS, INC.

Quarterly LTM: Round 1 2008

PURGE DATA SHEET - "LOW FLOW" GROUNDWATER

Site Name: Rubble Disposal Area, NAS South Weymouth
Sample ID: RDA-GW- -0408

Tetra Tech NUS Charge No. 112G00864 / CTO 407
QC: _____ (If applicable)

Clock Time 24hr	Water Depth below MP ft	Pump Dial 1	Purge Rate ml/min	Cum. Volume Purged Gals.	Temp °C	Spec. Cond. 2 uS/cm	pH (S.U.)	ORP/Eh3 mv	DO mg/L	Turbidity NTU	Comments
Acceptance Criteria: <0.3 ft (drawdown)					3%	3%	+/- 1.0 S.U.	+/- 10mV	10%	10%	

TtNUS Form 0009

Saturated Screen Volume (gallons) _____ (2" screen = 0.163 gals/ft of depth; 4" = 0.653 gals/ft; 6" = 1.469gals/ft)

1. Pump dial setting (for example: hertz, cycle/min, etc.)
2. Siemens per cm (same as umhos/cm) at 25 °C.
3. Oxidation reduction potential (stand in for Eh).

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Project Site Name: _____ Sample ID Number: _____
Project Number: _____ Sampled By: _____
Sample Location: _____ C.O.C. Number: _____
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other _____

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: _____
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:
[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
Volatiles	Cool 4°C & HCl		YES / NO
Semivolatiles	Cool 4°C		YES / NO
Pesticide / PCB	Cool 4°C		YES / NO
Metals	Cool 4°C & HNO_3		YES / NO
Cyanide	Cool 4°C & NaOH		YES / NO

OBSERVATIONS / NOTES:

Signature(s):

TETRA TECH NUS, INC.

SAMPLE COLLECTION SUMMARY RECORD

PROJECT NAME: _____ TETRA TECH NUS CHARGE NUMBER:_____

SAMPLING EVENT:_____ CASE NO.: _____ DAS NO.:_____

DATE	TIME	SAMPLE LOCATION	FIELD QC							COMMENTS

TtNUS Form 0012

TETRA TECH NUS, INC.

SITE ENTRY LOG

Site Name: _____ Date: _____

Location: _____ Project Number: _____

NAME	REPRESENTING	TIME IN (HOURS)	TIME OUT (HOURS)	INITIALS

TtNUS Form 0002

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date: / Time: / Method: / Monitor Reading (ppm):	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

Tetra Tech NUS, Inc.

SURFACE WATER SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

Date: / Time: / Depth: / Method:	**Color** (Visual)	**pH** (S.U.)	**S.C.** (mS/cm)	**Temp.** (^{0}C)	**Turbidity** (NTU)	**DO** (mg/l)	**Salinity** (%)	Other

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

TETRA TECH NUS, INC.

WELL INSPECTION AND GROUNDWATER LEVEL MEASUREMENT SHEET

WELL NUMBER: _____ PROJECT NAME: _____

DATE/TIME: _____ PROJECT MANAGER: _____

INSPECTED BY: _____

_____

VENT WELL

MONITORING INSTRUMENT READING: _____

LEL/02 READING: _____

WELL INSPECTION/GROUNDWATER LEVEL MEASUREMENT

WELL DEPTH (FEET FROM TOP OF PVC) _____

WATER LEVEL DEPTH (FEET FROM TOP OF PVC) _____

WELL STICK-UP _____

CASING STICK-UP (FEET) _____

WELL DIAMETER (INCHES) _____

WELL CONSTRUCTION (PVC, STEEL, ETC.) _____

LOCKED UPON ARRIVAL?	YES	NO
LOCKED REPLACED?	YES	NO
OBSTRUCTIONS?	YES	NO
WELL RELABELED?	YES	NO
SLUG TEST CONDUCTED?	YES	NO (If YES, refer to "Hydraulic Conductivity Testing Data Sheet")

GENERAL CONDITION/COMMENTS:_____
_____
_____
_____
_____

TETRA TECH NUS, INC.

YSI MULTIPARAMETER WATER QUALITY METER

Site Name: Naval Weapons Industrial Reserve Plant, Bedford, MA; Job No: 112G00893 / Semi-Annual Event

Serial No. _____ Model No. _____ Decal Letter _____;

Instrument is calibrated in accordance with manufacturers instructions

DATE:	Post Calibration Readings	PM Check	Calibration STDs	Signature	Remarks
Cond.= 1 mS/cm			Lot No. Exp.		
Cond. Check (DIUF)			Lot No. Exp.		
pH=4.0			Lot No. Exp.		
pH=7.0			Lot No. Exp.		
pH=10.0			Lot No. Exp.		
D.O. mg/l			% moisture in air		
REDOX mV			Lot No. Exp.		
Temp °C					

DATE:					
Cond.= 1 mS/cm					
Cond. Check (DIUF)					
pH=4.0					
pH=7.0					
pH=10.0					
D.O. mg/l					
REDOX mV					
Temp °C					

Mod. TtNUS Form 0024

APPENDIX C

PROJECT SPECIFIC FIELD TASK PROCEDURES

APPENDIX C-1

PROJECT-SPECIFIC SOIL SAMPLING PROCEDURES

Appendix C-1
Project-Specific Soil Boring and Sampling Procedures
Coddington Cove Rubble Fill Area Site
NAVSTA Newport, Newport, Rhode Island

Project-specific procedures are presented below for the collection of soil samples from Drive and Wash soil borings (including procedures for volatile and non-volatile analytical parameters and jar headspace screening). The SOPs and field documentation forms referenced below are included in Appendices A and B, respectively. The field team will follow the project-specific field procedures unless these procedures do not provide guidance on a specific field task issue. In that case, the procedures in the cited SOPs will be followed.

Preliminary sample locations are presented on Figure 17-1.

Soil Sample Collection from Soil Borings

Soil borings will be advanced at a total of eight locations, all of which will be completed as overburden groundwater monitoring wells.

Continuous split-barrel soil samples will be retrieved at 2-foot intervals from each of the eight boring locations. The samples will be logged and screened for VOCs using jar-headspace methods, and four samples from each boring location will be selected for laboratory analysis for a total of 32 samples to be analyzed. One of the four selected samples will be from the surface interval; samples from this interval will be collected from the 0-1 foot portion of the interval in accordance with USEPA Region I guidance for human health risk assessment. The second sample selected will be from the interval at the top of the bedrock or from the deepest interval sampled. The other two samples will be selected from intermediate depths by the geologist, with the assistance of the FOL, based on the headspace screening results, water table location, confining layers, and visual or olfactory evidence of contamination. This vertical sample allocation is designed to ensure complete spatial coverage of the soil overburden with an emphasis on sampling any intervals that field conditions indicate could be contaminated.

The selected soil samples and associated QC samples will be analyzed for VOCs, PAHs, pesticides, PCBs, and TAL metals. The analytical methods for these analyses are presented in Worksheet # 19. Worksheet # 15 lists target analytes, the SASE project action levels, and the project quantitation limits that must be met by the laboratory.

Drilling Procedures

Drilling will be performed by a subcontractor according to the procedures in SOP GH-1.3 (Appendix A). TtNUS will develop a scope of work, procure the subcontractor, and oversee all drilling activities. All down-hole drilling equipment will be steam-cleaned before use at each boring. Borings will be advanced to refusal (depth of borings estimated to be approximately 40 feet) using drive and wash drilling with driven casing to allow for 2-inch inside diameter (ID) wells to be installed. Continuous split-barrel samples will be retrieved at 2-foot intervals throughout the length of the boring using a 2-inch ID, stainless steel split-barrel sampler, and all required information will be recorded on the boring log sheet (Appendix B) according to SOP GH-1.5. The bedrock depth is unknown but is believed to range from 20 to 40 feet at the Site. No bedrock coring will be conducted.

Soil Sampling Procedures for Volatile Laboratory Samples (Grab)

As the split-barrel samplers are retrieved from the borehole, soil samples will be collected in accordance with SOP SA-1.3 (Appendix A). The split-barrel sampler will be opened, visually inspected, and scanned for VOCs using a photo-ionization detector (PID) according to SOP ME-12 or a flame-ionization detector (FID) according to SOP ME-15 (Appendix A). Grab samples will be collected from the most heavily contaminated portion of the split-barrel sampler, based on the initial screening results and/or visual observations. One sample aliquot will be used for jar headspace screening analysis (see procedures below), and a second aliquot will be collected according to the sampling procedures below for volatile samples and stored temporarily for possible laboratory analysis of VOCs. The specific depth of the grab volatile samples within the 2-foot split barrel sample will be recorded. The remainder of the soil interval will be kept for possible laboratory analyses for the non-volatile parameters.

After all split-barrel samplers have been recovered from a borehole and volatile samples collected, sample intervals will be selected for laboratory analysis based on the jar headspace screening results and other factors. The remaining contents of each selected interval will be uniformly mixed to form a composite sample and split into aliquots for the non-volatile analyses.

The procedures for collection of the volatile grab samples, jar-headspace screening, collection of a VOCs percent moisture sample, and collection of non-volatile composite samples are presented below.

<u>Soil Sampling Procedures for Volatile Laboratory Samples (Grab)</u>

Each soil sample for VOC analysis will be collected using a cut syringe or equivalent device and placed in two sodium bisulfate ($NaHSO_4$)-preserved (1 gram [g] $NaHSO_4$ in 5 milliliters [mL] reagent grade water)

vials with septa caps and one methanol-preserved vial with a septa cap, according to SW-846 Method 5035A (July 2002). The sodium bisulfate-preserved and methanol-preserved vials will be maintained at 4±2°C for analysis within 14 days. The following procedures shall be followed for the soil VOC sample collection:

1. Label two pre-tare weighed 40-mL amber vials containing 5 mL of reagent grade water (sodium bisulfate-preserved) and one 40-mL amber vial containing 5 mL of methanol with the sample location number and a bottle letter such as A, B, etc.

2. Collect approximately 5 grams of sample by coring or stabbing the soil with a 10-mL pre-cut syringe. If non-aqueous phase liquid (NAPL) is noted within the soils, then a reduced volume of approximately 1 to 2 grams should be collected as a separate "medium concentration" (NAPL) sample. Extrude the sample into one of the 40-mL VOC vials containing 5 mL of reagent grade water or methanol. The extruded soil must be immersed in the water or methanol; recollect the sample using a smaller volume, if necessary. Avoid touching the threads on the vial's neck or loss of water by evaporation. Cap the vial and invert it at least five times to mix the sample.

3. Weigh each sample vial to the nearest 0.01 gram and record the weight on the field log sheet. Repeat the sample collection procedure for the remaining vials. Ensure that the samples are properly labeled, maintained in coolers with ice, and that the chain-of-custody procedures described in Worksheet #27 are followed. Package and prepare for pickup or ship the sample coolers to the appropriate laboratory for overnight delivery. Include the field log sheet containing the sample weight information with the samples.

Field duplicate samples will also be collected. Following the collection of the first set of VOC containers, collect the field duplicate **from the same sampling interval as the original sample.**

For laboratory QC analyses, collect six sodium bisulfate-preserved vials and three methanol preserved vials of soil samples.

Jar Headspace VOC Screening Procedures

An aliquot of soil will be collected for jar headspace screening with a PID or FID for all soil sample intervals. The procedure for the headspace screening is provided below:

1. Collect sufficient soil representative of the sample interval to half-fill one clean 8-oz. glass jar. Quickly cover the jar with clean aluminum foil and apply the screw cap ring (without lid) to tightly seal the jar.

All appropriate analytical sampling procedures should be followed to maintain this sample matrix as representative and to avoid cross-contamination.

2. Vigorously shake the jar for 15 seconds. Allow headspace development for at least 10 minutes. Headspace development shall be performed within a vehicle or building that is temperature controlled between 65°F and 80°F. Ambient temperatures during the screening tests will be recorded with the field notes.

3. Quickly puncture the foil seal with the instrument probe, and advance the probe tip to about one-half of the headspace depth. Exercise care to avoid the uptake of water droplets or soil particles.

4. Record the highest instrument reading as the jar headspace VOC concentration. The maximum response should occur between 2 to 5 seconds. Erratic meter response may occur with high organic vapor concentrations or high moisture content. If erratic responses are obtained, stop the headspace screening.

5. Operation, maintenance, and calibration of the monitoring instrument will be performed in accordance with SOP ME-12 (Photovac 2020 PID) and ME-15 (Photovac Micro FID), as applicable. The SOPs are included in Appendix A.

Soil Sample for Percent Moisture

Fill one 2-oz. container with soil representing the same locations where the 40-mL VOC sample vials were collected. Every effort should be made to obtain the percent moisture soil aliquot as close as possible to the location where the VOC samples were collected.

Soil Sampling Procedures for Non-Volatile Parameters

1. Record all required data on the boring log, which will also serve as the soil sample log sheet (Appendix B). Include the sampling equipment, sampling personnel, date, time, depth of sample, and sample analyses. The boring log will also contain soil descriptions, depth of strata changes, and sample depth intervals. The soil will be visually classified using the Unified Soil Classification System (ASTM D-2488-98).

2. Label appropriate sample jars with the sample location number, sampler's name, date, and analytical fractions.

3. Transfer the soil from the split-barrel sampler to a decontaminated stainless-steel bowl using only decontaminated stainless steel trowels, and homogenize the sample.

4. If there is insufficient sample volume to fill all the containers for the non-VOC analyses, an equal amount of materials from the intervals immediately above and below the selected sample interval may be used to supplement the composite sample to ensure sufficient sample quantity for all analyses.

5. Remove any large particles such as gravel or artificial fill too large to be sent for analysis. Note the removal of material on the boring log.

6. Fill the appropriate sample containers.

7. For field duplicate samples, after homogenization, fill one set of sample containers for the original sample and fill another set for the field duplicate sample.

8. Ensure that the samples are properly labeled, maintained in coolers with ice, and that the COC procedures described in Section 14.3.3 are followed. Package and ship the sample coolers to the appropriate laboratory for overnight delivery.

9. Decontaminate the sampling equipment before reuse.

Care should be taken in handling all soil samples to ensure that the exterior of the sample containers are clean and free of soils before shipping to the laboratory.

Drill cuttings and excess soil samples will be contained in drums and stored on-site for characterization and disposal.

APPENDIX C-2

PROJECT-SPECIFIC PROCEDURES FOR WATER LEVEL MEASUREMENT AND GROUNDWATER SAMPLING

Appendix C-2

Project-Specific Procedures for Well Completion and Development, Water Level Measurement, and Groundwater Sampling

Coddington Cove Rubble Fill Area

NAVSTA Newport, Newport, Rhode Island

Project-specific procedures for monitoring well completion and development, water level measurement, and groundwater sampling are presented below. The SOPs referenced below are included in Appendix A. The Field Team will follow the project-specific field procedures unless these procedures do not provide guidance on a specific field task issue. In that case, the procedures in the cited SOPs will be followed.

Monitoring Well Completion

The drilling subcontractor will install monitoring wells within the boreholes at each of the eight boring locations (Figure 14-1). The wells will be constructed according to SOP GH-2.8 (Appendix A) using 2-inch ID, Schedule 40 polyvinyl chloride (PVC) casing with threaded joints and 10-slot (0.010-inch) screened PVC threaded riser. In general, wells will be screened across intervals of interest identified by the field geologist. Wells screened in the shallow overburden will have 10-foot screens and will be installed with the bottom of the screen approximately 5 feet below the water table. Deeper, non-water table wells will also be constructed with a 10-foot screen. However, under certain conditions (e.g. a potentially contaminated zone bracketed by less-transmissive zones) a shorter screen may be used.

The appropriate specification for the filter pack and well screens will be determined by the field geologist after review of the prevalent geologic conditions. At least two different filter pack materials will be available at the site during well installation activities.

Upon completion of the boring, well screen and riser will be assembled with a silt-trap on the bottom and placed in the borehole. A filter pack will be installed in the annular space from the bottom of the hole to a depth of 2 to 4 feet above the top of the screen. To ensure proper placement of the filter pack within the screen interval (i.e., minimize voids and the potential for pack consolidation and grout contamination), the well may be surged using a surge block after the sand is delivered to the screen interval. The intent of this procedure is to minimize the collapse of soil that prevents the uniform distribution of sand. The quantity of sand recovered during surging will be monitored to determine the most efficient technique at each well location.

The filter pack material will consist of a clean quartz silica sand meeting American Water Works Association (AWWA) Standard B-100-89 for purity, composition, size distribution, and uniformity for "00S" class sand. A 1 to 2 foot thick bentonite seal will then be placed immediately above the filter pack, and the remaining annular space will be backfilled with grout (a cement-bentonite mixture). If the top of the sand layer is less than 10 feet below ground surface, the driller may elect to backfill the borehole with hydrated bentonite instead of grout. Monitoring wells will be completed with elevated (stick-up) protective casings in all areas. A cement apron will be installed to hold the protective casing in place. The well installation will be documented on a monitoring well construction log sheet (Appendix B).

Well development will be conducted no earlier than 3 days after installation to allow seals and other installations to set.

Monitoring Well Development

After installation of the new groundwater monitoring wells and prior to groundwater sampling, the eight monitoring wells (Figure 14-1) will be developed by surging and pumping. Well development is conducted to ensure that the aquifer is hydraulically connected with the well. Proper well development is necessary to:

- remove silt and other fine material from the well and filter pack;
- establish good hydraulic connection with the formation;
- restore the permeability of the formation adjacent to the filter pack; and
- ensure collection of representative samples of groundwater.

Development will be conducted according to the procedures in SOP GH-2-8 (Appendix A). This will include alternately surging the well with a surge block and removing water from the well using submersible pumps operating at various flow rates or by bailing. Measurements of pH, specific conductance, temperature, dissolved oxygen, and turbidity will be periodically collected during development and recorded in the logbook. The pH, specific conductance, and temperature will be measured with a water quality instrument. Turbidity will be measured separately with a nephelometer. Development will proceed until the extracted water is clear or for a maximum of 4 hours. Wells that dry up during development due to a slow recharge rate will be considered developed once the well has be evacuated of all water at least three times after a minimum of 90% recovery of the static water level. Any well that does not produce clear water at the conclusion of well development will be noted in the field logbook.

All development results will be recorded in well development data sheets (Appendix B) and evaluated to determine if any of the wells have turbidity and/or low-flow problems. A general review of some possible problems and recommendations to address them are provided below.

High Turbidity Values

Raise the bottom of dedicated tubing
Further development and purge testing
Installation of replacement monitoring well

Low Purging and Sampling Flow Rate

Further development and purge testing
Exceed maximum drawdown (0.3 ft) if turbidity remains acceptable
Collect sample at low flow rate

Purged groundwater will be captured in drums and stored on-site for characterization and disposal.

The water levels in the wells will be allowed to stabilize for 5 days or more after development, depending on the permeability of the formation, prior to groundwater level measurement and sampling.

Water Level Measurement

Groundwater level measurements will be recorded at the eight newly-installed groundwater monitoring wells in order to determine the gradient and estimate the direction of groundwater flow at the Site. Measurements will be taken at all of the wells in as short a period of time as possible in order that the water levels are not affected by changing weather conditions.

The groundwater level measurements will be obtained according to the procedures in SOP GH-1.2 (Appendix A) using an electronic water level indicator with a weighted cord that is accurate to 0.01 feet. Water levels will be measured from the highest point of the top of the PVC riser (marked by the FOL) and recorded on a groundwater level measurement form (Appendix B).

Groundwater Sampling

Groundwater samples will be collected at the eight newly-installed and developed monitoring wells (Figure 14-1) for laboratory analysis. In addition, QC samples will be collected for analysis as part of the groundwater investigation, including field duplicates, rinsate blanks, field blanks, and laboratory QC samples. QC samples are described in Worksheet # 20.

All groundwater and QC samples will be analyzed for VOCs, SVOCs, pesticides, PCBs, and total and dissolved TAL metals. Worksheet # 15 lists target analytes, the SASE project action levels, and the project quantitation limits that must be met by the laboratory. The analytical methods, types of sample containers, preservatives, and holding times for each analytical group are presented in Worksheet #19.

All monitoring wells will be sampled using low stress (low-flow) purging and sampling procedures according to the GW-001 EPA Region I low-flow SOP (Appendix A). This method emphasizes the need to minimize water-level drawdown and groundwater pumping rates in order to collect samples with minimal alterations to groundwater chemistry.

Monitoring wells will be checked for the presence of NAPL using an oil water interface probe and bailer prior to initiating the purging process. If NAPL is detected, a sample of the NAPL will be collected.

Groundwater Sampling Procedures

The pumping system will be designed to be consistent with the intent of the low-flow groundwater sampling method. Dedicated tubing will be used for each monitoring well to minimize cross-contamination between monitoring wells. Filtered and unfiltered samples will be collected for metals analysis using a 0.45 micron in-line filter. One rinsate blank for metals analysis will be collected from the filtration system.

During well purging, water level drawdown and flow rate will be recorded on low-flow groundwater sample log sheets (Appendix B). Groundwater will be pumped through a flow-through cell and the pH, specific conductance, temperature, DO, ORP, and salinity will be measured with a water quality instrument and recorded. Turbidity will be measured separately with a nephelometer. The instruments will be calibrated according to the manufacturer's specifications as discussed in Section 14.1.13. Purging is considered complete and sampling may begin when all parameters

have stabilized. Stabilization is considered to be achieved when three consecutive readings, taken at 3- to 5-minute intervals, are within the following limits:

Turbidity (less than 5 Nephelometric Turbidity Units (NTU); or if turbidity greater than 5 NTU, readings within + or – 10%);
DO (+ or – 10% for values greater than 2 mg/L; within 0.5 mg/L for values less than 2 mg/L);
Specific conductance (+ or – 3%);
Temperature (+ or – 3%);
pH (+ or – 0.1 units);
ORP (± 10 millivolts); and
Drawdown (no more than 0.3 feet).

If stabilization has not occurred within two hours sampling can begin and a note will be made on the low-flow sample log sheet that stabilization was not achieved.

All pertinent sampling information will be recorded in the field logbook and on a low-flow groundwater sample log sheet (Appendix B). Salinity will be noted on the chain-of-custody form (Appendix B) in order to aid the analytical laboratory in establishing dilution requirements for metals analyses where salinity could present an interference concern.

Purged groundwater will be captured in drums and stored on-site for characterization and disposal as described in Section 14.1.13.

The sample collection documentation, handling, tracking, and custody procedures are presented in Worksheet #27.

Worksheet #18 presents the analytical field program summary. Worksheet #19 presents the analytical methods, sample container types, preservative requirements, and the maximum holding times for the associated analyses. Worksheet #20 presents the quality control sample summary. The QC task is also described in Worksheet #14.

APPENDIX D

ANALYTICAL SPECIFICATION

ATTACHMENT NO. 2

STATEMENT OF WORK/PRICE TABLES

TECHNICAL SPECIFICATION FOR LABORATORY SERVICES
SITE 4, CODDINGTON COVE RUBBLE FILL AREA
NAVAL STATION NEWPORT, NEWPORT, RHODE ISLAND

COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION - NAVY (CLEAN)
CONTRACT N62470-08-D-1001, CONTRACT TASK ORDER (CTO) NO. WE48

STUDY AREA SCREENING EVALUATION
CHEMICAL ANALYSES

1.0 INTRODUCTION

Tetra Tech NUS, Inc. (TtNUS) under CLEAN Contract N62470-08-D-1001, is procuring laboratory analytical services to support a Study Area Screening Evaluation (SASE) at Naval Station (NAVSTA) Newport, Site 4, Coddington Cove Rubble Fill Area. Requested analyses include volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), polycyclic aromatic hydrocarbons (PAHs), pesticides, polychlorinated biphenyls (PCBs), gasoline range organics (GRO), diesel range organics (DRO), and target analyte list (TAL) metals.

The laboratory performing these analyses must be certified in the State of Rhode Island. Additionally, the laboratory must provide a copy of its Naval Facilities Engineering Services Center (NFESC) approval letter or Department of Defense (DOD) Environmental Laboratory Accreditation Program (ELAP) accreditation letter; all methods requested must be addressed.

The responding laboratory must submit method detection limits (MDLs), Limits of Detection (LODs), and Limits of Quantitation (LOQs) for all analyses and matrices requested by filling out the last two columns of the tables in Attachment A and including the completed attachment with the bid response. After award, the laboratory will be required to submit Standard Operating Procedures (SOPs) and relevant precision and accuracy limits for all preparation and analytical methods required under this scope of work. The laboratory will also be asked to complete Uniform Federal Policy (UFP) Sampling and Analysis Plan (SAP) Worksheets 19, 23-26, and 28 for inclusion in the SAP. The SAP will be prepared according to the UFP for Quality Assurance Project Plans (March 2005) and will utilize the 37 UFP-SAP worksheets.

2.0 SAMPLE INFORMATION

The approximate number of samples to be submitted, the type of analyses to be conducted, and the analytical methods to be used are summarized in Table 1. This SASE includes analysis of groundwater, soil, sediment, and surface water samples.

The sampling is scheduled for several weeks in October 2010. The exact date of initial sample collection will be communicated to the laboratory at least two weeks in advance.

The samples are expected to be of low or moderate contaminant concentration. The field crew will attempt to identify any potentially high concentration samples.

Field duplicate samples will be submitted to the laboratory with "blinded" identification. The field crew will designate samples (one per twenty samples of like matrices) for matrix spike/matrix spike duplicate (MS/MSD) (organic analyses) or matrix spike/laboratory duplicate (metals). Additional volumes of these samples will be provided as necessary.

For the sediment non-volatile analyses, the laboratory must decant any standing water, homogenize the sample, and determine the percent moisture before sample analysis. The non-volatile sample aliquots must be increased to compensate for the moisture content of the samples. The PALs listed in Attachment A must be met regardless the moisture content of the sediment samples.

If the percent moisture content is too high and the sediment samples contain noticeable organic material, further dewatering must be performed at the laboratory prior to sample analysis. This could be accomplished by freeze-drying under controlled conditions proven to recover the analysis-specific surrogates; centrifugation and decanting free water; low temperature oven drying (below 60°C); or other procedure proposed by the laboratory and approved by TtNUS.

Soil and Sediment Volatile Analyses

Soil and sediment samples for VOC and GRO analysis will be collected using a coring device (cut off syringe). The following aliquots will be collected:

- Two 40-ml VOA vials pre-preserved with 1 g NaHSO4 in 5 ml VOC-free reagent water w/ a magnetic stir bar.
- Three 40-ml VOA vials pre-preserved with 5 ml of methanol (one for VOCs, two for GRO)
- One 2-oz wide-mouth jar for percent solids

The pre-preserved VOC and GRO sample containers must be weighed accurately to within 0.01 grams and identified with a unique ID number. Both the ID number and the applicable vial weight must be recorded on a weight tracking form for return shipment to the laboratory. When samples are received at the laboratory, the pre-preserved vials must be re-weighed and these values recorded in the weight tracking form. TtNUS must be contacted immediately if leaking vials are received at the laboratory.

The VOC low-concentration analysis (bisulfate-preserved vials) must be performed first for all of the soil and sediment samples in order to meet the required project action limits (PALs). If any target compound is above the calibration range, the laboratory should perform a dilution analysis using a methanol-preserved vial, and the medium level (methanol-preserved) trip blank associated with that sample must also be analyzed.

GRO/DRO Analyses

The GRO fraction must include all petroleum hydrocarbons ranging from **C_5 to C_{12}**. The diesel range organic analysis (DRO) must be extended to include the **C_9 to C_{36}** petroleum hydrocarbons. In addition to quantitating the GRO and DRO concentrations, the laboratory must identify the types of fuel contamination present in the samples. The gas chromatograms must depict the fingerprint patterns at greater than 25 percent of the full scale.

Groundwater SVOC Analysis

SVOC full scan or SIM analyses of groundwater samples are specified for the list of compounds in Attachment A. If any compound concentration required to be analyzed by SIM exceeds the upper instrument calibration level and an acceptable result is obtained from full scan analysis, there is no need to perform dilutions for the SIM analysis; the result may be reported from the full scan analysis.

Pesticide and PCB Analyses

All positive identifications for pesticides and PCBs by gas chromatography (GC) with electron capture detector (ECD) MUST be confirmed on a second column that possesses retention characteristics different from those exhibited by the primary column. Identification using a single column with dual detectors does not meet second column confirmation requirements. Confirmed positive results less than the laboratory LOQ but greater than the MDL must be reported by the laboratory; the laboratory must "J" flag these results. The higher of two results generated from dual column analyses must be reported as the primary result by the laboratory unless relative percent differences (RPDs) between columns are greater than 40% and interference is found to be present or there is some other justifiable reason to select the lower of the two results. The laboratory must narrate the reason for selection of the lower result.

Metals Analyses

For the ICP and mercury metals analyses, perform a matrix spike and a laboratory duplicate analysis instead of matrix spike/matrix spike duplicate. If an ICP metals matrix spike recovery falls outside the control limits and the sample result does not exceed four times the spike added, a post-digestion spike analysis must be performed. In addition, a five-fold dilution analysis (ICP serial dilution) must be performed on the field-designated QC sample. Results for these QC analyses must be summarized in the corresponding CLP-equivalent forms (Attachment B).

3.0 ANALYSIS/REPORTING INFORMATION

One hard copy data package deliverable and two PDF CD copies must be submitted, in addition to the electronic data deliverables to be provided in the format described in Attachment C. The original chain-of-custody form received with the samples and signed by the laboratory sample custodian must be returned with the hard copy data package.

The analytical requirements and approximate number of samples to be submitted for analysis are detailed in Table 1. Analysis and reporting requirements addressed in the Department of Defense (DOD) Quality Systems Manual (January 2006 or April 2009 depending on current NFESC approval letter) and the requested methods must be followed. Additionally, it is a requirement of TtNUS that the associated PDF and hard copy data packages for VOC, SVOC, PAH, Pesticide, PCB, and metal analyses must meet Contract Laboratory Program (CLP) format, reporting, and PDF/hard copy data package deliverable requirements. The PDF and hard copy data packages for the GRO and DRO analyses must be in a CLP-modified format and must include the appropriate summary forms and raw data for all samples and laboratory quality control samples. The summary forms should include the method-specific quality control limits (recoveries, relative percent differences, relative standard deviations, and/or percent differences, etc.). The TtNUS sample identification numbers **must be included** on the raw data and summary forms.

Additionally, each hard copy and PDF data package must contain a summary data package. This summary data package shall consist of only the summary forms (i.e., for CLP, Forms 1 through 15; for non-CLP it shall be the CLP-like equivalent of Forms 1 through 15). Attachment B details the required summary forms for CLP-like data packages and requirements for organization/bookmarking of hard copy/PDF data packages.

Attachment A details the required target compound list and required PALs that must be met. The laboratory must submit its LOQs, LODs, and MDLs for all analyses and matrices requested by filling out the last two columns of the tables in Attachment A and including the completed attachment with the bid response. If the PAL for a target compound is not technically achievable, or if no PAL is listed for a target compound in Attachment A, the laboratory should propose the lowest LOQ technically possible for the requested method.

Non-detected organic and metals results results must be reported down to the laboratory LODs. Positive results above the MDL but below the laboratory's LOQ must be reported as estimated values qualified with a "J" **for both organic analyses and metals** analysis. Soil samples must be reported on a dry-weight basis.

The hard copy/PDF data package deliverable must contain a detailed case narrative for all analytical fractions. This case narrative must also include the Contract Task Order (CTO) number, the site name, and the TtNUS Project Manager's name. Data from all analytical runs (i.e., original, dilution, re-analysis) must be reported in the raw data and Form Is for organic analyses. For metals analyses, only the final sample results should be reported in the Form Is, and data from all analytical runs must be included in the raw data.

As part of the laboratory case narrative, it is required that the Laboratory Quality Assurance Manager sign an attestation statement verifying that all electronic diskette deliverables exactly match the data summary forms (i.e. Form Is).

As stipulated in the CLEAN Master Services Agreement (MSA), Sample Delivery Group (SDG) and fractionally-specific text (TXT) files containing all environmental sample and field quality control blank analysis results must be generated in accordance with the requirements outlined in Attachment C of this specification.

Maximum holding time allowances, as defined in the following table, are to be strictly observed. Calculation of holding time is in calendar days and is to begin from the time of sample collection. The holding times are as follows:

Analyses	Preservation	Holding Time
VOCs	Aqueous: HCL to pH < 2, cool to 4° C Solid: 1 g $NaHSO_4$ in 5 ml reagent water to pH < 2, cool to 4°C; or 5 ml methanol, cool to 4°C	Aqueous and Solid: 14 days to analysis
GRO	Aqueous: HCL to pH < 2, cool to 4° C Solid: 5 ml methanol, cool to 4°C	Aqueous and Solid: 14 days to analysis
SVOCs, PAHs, Pesticides, PCBs, DRO	Aqueous and Soil: Cool to 4° C	Aqueous: 7 days to extraction, 40 days to analysis Solid: 14 days to extraction, 40 days to analysis
Metals (total and dissolved*)	Aqueous: HNO_3 to pH < 2, cool to 4° C Soil: Cool to 4° C	Aqueous and Solid: 6 months to analysis for ICP metals; 28 days for mercury

* Samples will be filtered in the field.

These holding times are based on 40 CFR 136, data validation criteria, and method specific requirements, and are measured from date of collection for samples preserved as requested in the analytical methods. The holding time criteria depicted apply to all analyses necessary to successfully determine the contaminant level contained in the sample. Hence, **the holding time criteria apply to any/all subsequent sample dilutions and re-analyses**.

The TtNUS Project Manager for this project is Mr. Thomas Campbell and he must be contacted in the event of any laboratory problems that could impact project deadlines (i.e., late deliverables, technical problems in the lab that could lead to late deliverables.) To ensure good communication, it is required that the laboratory's appointed project manager contact Mr. Campbell once a week for the entire project duration.

Contact information for Mr. Campbell is as follows:

Tetra Tech NUS
55 Jonspin Road
Wilmington, MA 01887
Phone: 978-474-8404
Fax: 978-474-8499
Email: thomas.campbell@tetratech.com

Analytical data turnaround times are to be measured from receipt of each sample shipment. All hard copy/PDF (2 CDs) analytical data packages and associated electronic (TXT) deliverables are due within the standard MSA turnaround term of 21 calendar days from receipt of the last sample in a Sample Delivery Group (SDG).

The SDGs must contain 20 samples, unless enough samples to complete the SDG are not anticipated to be received within a time period that will allow analysis within holding time. The frequency with which SDGs contain less than 20 samples should be minimal. All PDF data packages and electronic deliverables must be received at the same time or the deliverable will be considered incomplete and payment deductions may be imposed.

The hard copy/PDF (2 CDs) analytical data package and the original chain-of-custody form (received with the samples and signed by the laboratory sample custodian) should be sent to Ms. Lucy Guzman. Contact information for Ms. Guzman is the same as noted above for Mr. Campbell except that her direct phone number is (978) 474-8416 and her email address is lucy.guzman@tetratech.com.

The electronic (TXT) deliverables, in addition to a copy of the chain-of-custody form, should be sent to Ms. Tobrena Skeen. Contact information for Ms. Skeen is as follows:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220-2745
Phone: 412-921-8582
Fax: 412-921-4040
e-mail: tobrena.skeen@tetratech.com

4.0 PERIOD OF PERFORMANCE/BOTTLEWARE INFORMATION

All samples will be shipped to the laboratory via express carrier within 48 hours of collection. Please circle the Yes or No at the bottom of Table 1 which will indicate if the laboratory will provide courier service at no extra charge. The laboratory will be notified at least 2 weeks prior to sample collection.

Bottleware shipments will be coordinated by the field operation leader.

The laboratory is to provide all necessary sample containers **(plus approximately 10% extra for breakage)**. All sample containers must meet ICHEM series 300 cleanliness criteria (or equivalent), and documentation of certified cleanliness must be provided. All of the appropriate sample bottleware must be pre-preserved. The bottleware must be shipped to the designated location in Coleman-like coolers. Each cooler must include a "temperature blank" vial. **The laboratory must also provide any extra coolers needed for return shipment of samples to the laboratory for analysis.** The laboratory is also requested to provide a packing slip indicating the analytical parameters for which each container type is designated, sample labels, and chain-of-custody forms.

For this project, TtNUS will analyze 46 soil samples for VOCs and GRO; however, **the laboratory is requested to provide additional sets of pre-preserved VOC and GRO sample containers for 128 soil interval samples to be collected but not analyzed.** The laboratory should include the cost of these additional pre-preserved sample containers as a separate line item in the price sheet (Table 1).

The laboratory must provide Material Safety Data Sheets (MSDSs) for all preservatives sent with each bottleware shipment to the field. MSDSs must be representative of the chemicals provided as preservatives with regard to mixtures and/or purity of the chemicals. For example if a 35% sulfuric acid solution is the preservative, the MSDS provided should be for 35% sulfuric acid solution not 96% sulfuric acid.

5.0 ADDITIONAL COMMENTS/CONTACTS

Within the laboratory, the internal transfer of samples, extracts, and digestates must be accomplished and documented as controlled custody transfers. The laboratory must submit the documentation that supports an unbroken chain of custody for samples, digestates and extracts from time of receipt or production in the laboratory until disposal.

The laboratory is to provide a minimum of 60 days storage of sample extracts/digestates and 60 days storage of intact leftover sample aliquots, as stipulated in the MSA. **Additionally, the laboratory must store PDF data packages for 5 years.**

All analyses conducted under this subcontract assignment are to be performed at the solicited facility only. The laboratory is not permitted to lower-tier subcontract these analyses, or analyze these samples at a corporate facility other than the facility stipulated without prior notification and consent from the CLEAN Subcontracting Officer.

The unit cost for analysis is to include compensation for containers, preservatives, coolers, shipping costs, storage, disposal, and laboratory quality control analyses (such as matrix spike, matrix spike duplicate,

laboratory duplicate, and laboratory control sample analyses.) These items are not to be billed as separate line items.

Technical, quality assurance, and data format concerns are to be directed to Ms. Lucy Guzman at 978-474-8416 or via e-mail lucy.guzman@tetratech.com. Ms. Guzman must be contacted and informed of any difficulties encountered during the conduct of the requested analyses.

Contract concerns, and response to this solicitation, are to be directed to:

Ms. Meg Price
CLEAN Subcontracting Officer
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406-1433
Phone: 610-491-9688
Fax: 610-491-9645
e-mail: meg.price@tetratech.com

Triplicate copies of invoices associated with the analyses contracted herein are to be submitted to the attention of the Accounting Supervisor:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220
Phone: 412-921-8506
Fax: 412-921-4040

Please confirm the laboratory's ability to perform the methodologies requested at the analyte quantitation limits indicated. Also confirm available laboratory capacity, and complete/confirm the costing information indicated in Table 1. All costing information must reflect the terms and conditions established by the 2009 CLEAN MSA.

TABLE 1
NUMBER OF SAMPLES/ANALYTICAL METHODS
SITE 4, CODDINGTON COVE RUBBLE FILL AREA, NAVSTA NEWPORT, NEWPORT, RI
STUDY AREA SCREENING EVALUATION

Matrix	Parameter	Method	# Samples	Unit Price	Total Cost
Groundwater and Associated Field Blanks	VOCs	SW-846 5030/8260B	12	$	$
	GRO	SW-846 5030/ 8015B(1)	12	$	$
	DRO	SW-846 3510C or 3520C/ 8015B(1)	10	$	$
	SVOCs	SW-846 3510C or 3520C/ 8270C or 8270C SIM(2)	10	$	$
	Pesticides	SW-846 3510C or 3520C/8081A	10	$	$
	PCBs	SW-846 3510C or 3520C/8082	10	$	$
	TAL Metals (Total)	SW-846 3010B/ 6010B/7470A	10	$	$
	TAL Metals (Dissolved)(3)	SW-846 3010B/ 6010B/7470A	10	$	$
Soil and Sediment	VOCs	SW-846 5035/8260B	55	$	$
	GRO	SW-846 5035/ 8015B(1)	55	$	$
	DRO	SW-846 3540C or 3550B/ 8015B(1)	52	$	$
	PAHs	SW-846 3540C or 3550B/ 8270C SIM	52	$	$
	Pesticides	SW-846 3540C or 3550B/ 8081A	52	$	$
	PCBs	SW-846 3540C or 3550B/ 8082	52	$	$
	TAL Metals	SW-846 3050B/ 6010B/7471A	52	$	$
Surface Water and Field Blanks Associated with Surface Water, Soil, and Sediment Samples	VOCs	SW-846 5030/8260B	12	$	$
	GRO	SW-846 5030/8015B(1)	12	$	$
	DRO	SW-846 3510C or 3520C/ 8015B(1)	11	$	$
	PAHs	SW-846 3510C or 3520C/ 8270C SIM	11	$	$
	Pesticides	SW-846 3510C or 3520C/ 8081A	11	$	$
	PCBs	SW-846 3510C or 3520C/ 8082	11	$	$
	TAL Metals (Total)	SW-846 3010B/ 6010B/7470A	11	$	$
	TAL Metals (Dissolved)(3)	SW-846 3010B/ 6010B/7470A	8	$	$
Additional sets of pre-preserved VOC containers for 128 soil samples				$	$
Additional sets of pre-preserved GRO containers for 128 soil samples				$	$

(1) GRO range C5-C12; DRO range C9-C36. (2) PAHs, 1,4-dioxane, hexachlorobenzene, and pentachlorophenol by SIM. (3) Dissolved Metals samples will be filtered in the field.

TOTAL COST $

Can the laboratory provide sample pick-up on site? YES or NO (circle one)

If yes is there a charge and what is that charge?_____

Name of Laboratory_____

Signature_____

ATTACHMENT A

Required Target Compound Lists and Project Quantitation Limit Goals
(See worksheets for groundwater, soil, sediment, and surface water in accompanying Excel file)

ATTACHMENT A
GROUNDWATER REQUIRED TARGET COMPOUND LIST AND PROJECT QUANTITATION LIMIT GOALS
SITE 4, CODDINGTON COVE RUBBLE FILL AREA, NAVSTA NEWPORT, NEWPORT, RI

Groundwater Samples - Analyte	CAS Number	Analytical Method	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Laboratory-specific[1]		
						LOQ	LOD	MDL
VOCs (ug/L)								
1,1,1-Trichloroethane	71-55-6	8260B	200	MCL	66.7			
1,1,2,2-Tetrachloroethane	79-34-5	8260B	0.067	RSL	0.02			
1,1,2-Trichloroethane	79-00-5	8260B	5	MCL	1.7			
1,1-Dichloroethane	75-34-3	8260B	2.4	RSL	0.8			
1,1-Dichloroethene	75-35-4	8260B	7	MCL	2.3			
1,2,4-Trichlorobenzene	87-61-6	8260B	70	MCL	23.3			
1,2-Dibromoethane (EDB)	80-97-7	8260B	0.05	MCL	0.02			
1,2-Dibromo-3-Chloropropane (DBCP)	96-12-8	8260B	0.2	MCL	0.1			
1,2-Dichlorobenzene	95-50-1	8260B	600	MCL	200.0			
1,2-Dichloroethane	107-06-2	8260B	5	MCL	1.7			
1,2-Dichloropropane	78-87-5	8260B	5	MCL	1.7			
1,3-Dichlorobenzene	541-73-1	8260B	--	--	--			
1,4-Dichlorobenzene	92-87-5	8260B	75	MCL	25.0			
2-Butanone	78-93-3	8260B	7100	RSL	2367			
2-Hexanone	591-78-6	8260B	4.7	RSL	1.6			
4-Methyl-2-pentanone	108-10-1	8260B	200	RSL	66.7			
Acetone	67-64-1	8260B	2200	RSL	733.3			
Benzene	71-43-2	8260B	5	MCL	1.7			
Bromodichloromethane	75-27-4	8260B	80	MCL	26.7			
Bromoform	75-25-2	8260B	80	MCL	26.7			
Bromoethane	74-96-4	8260B	--	--	--			
Carbon disulfide	75-15-0	8260B	100	RSL	33.3			
Carbon tetrachloride	56-23-5	8260B	5	MCL	1.7			
Chlorobenzene	108-90-7	8260B	100	MCL	33.3			
Chloroethane	75-00-3	8260B	2100	RSL	700.0			
Chloroform	67-66-3	8260B	80	MCL	26.7			
Chloromethane	74-87-3	8260B	19	RSL	6.3			
cis-1,2-Dichloroethene	156-59-2	8260B	70	MCL	23.3			
cis-1,3-Dichloropropene	10061-01-5	8260B	0.43	RSL	0.1			
Cyclohexane	110-82-7	8260B	1300	RSL	433.3			
Dibromochloromethane	124-48-1	8260B	80	MCL	26.7			
Dichlorodifluoromethane	75-71-8	8260B	39	RSL	13.0			
Ethylbenzene	100-41-4	8260B	700	MCL	233.3			
Isopropylbenzene	98-82-8	8260B	68	RSL	22.7			
Methyl acetate	79-20-9	8260B	3700	RSL	1233			
Methylcyclohexane	108-87-2	8260B	--	--	--			
Methylene chloride	75-09-2	8260B	5	MCL	1.7			
Methyl-tert-butyl ether	1634-04-4	8260B	1.2	RSL	0.4			
Styrene	100-42-5	8260B	100	MCL	33.3			
Tetrachloroethene	127-18-4	8260B	5	MCL	1.7			
Toluene	108-88-3	8260B	1000	MCL	333.3			
trans-1,2-Dichloroethene	540-59-0	8260B	100	MCL	33.3			
trans-1,3-Dichloropropene	542-75-6	8260B	0.43	RSL	0.1			
Trichloroethene	86-42-0	8260B	5	MCL	1.7			
Trichlorofluoromethane	75-69-4	8260B	130	RSL	43.3			
Vinyl chloride	75-01-4	8260B	2	MCL	0.7			
Xylenes (total)	1330-20-7	8260B	10000	MCL	3333			

SVOCs (ug/L)								
1,4-Dioxane	123-91-1	8270C SIM	--	--	--			
1,1'-Biphenyl	92-52-4	8270C	180	RSL	60.0			
2,2'-Oxybis(1-chloropropane)	108-60-1	8270C	--	--	--			
2,4,5-Trichlorophenol	95-95-4	8270C	370	RSL	123.3			
2,4,6-Trichlorophenol	88-06-2	8270C	6.1	RSL	2.0			
2,4-Dichlorophenol	120-83-2	8270C	11	RSL	3.7			
2,4-Dimethylphenol	105-67-9	8270C	73	RSL	24.3			
2,4-Dinitrophenol	51-28-5	8270C	7.3	RSL	2.4			
2,4-Dinitrotoluene	121-14-2	8270C	0.22	RSL	0.1			
2,6-Dinitrotoluene	606-20-2	8270C	3.7	RSL	1.2			
2-Chloronaphthalene	91-58-7	8270C	290	RSL	96.7			
2-Chlorophenol	95-57-8	8270C	18	RSL	6.0			
2-Methylphenol (o-cresol)	95-48-7	8270C	180	RSL	60.0			
2-Nitroaniline	88-74-4	8270C	37	RSL	12.3			
2-Nitrophenol	88-75-5	8270C	--	--	--			
3,3'-Dichlorobenzidine	91-94-1	8270C	0.15	RSL	0.1			
3-Nitroaniline	99-09-2	8270C	--	--	--			
4,6-Dinitro-2-methylphenol	8071-51-0	8270C	--	--	--			
4-Bromophenyl phenyl ether	101-55-3	8270C	--	--	--			
4-Chloro-3-methyl phenol	59-50-7	8270C	--	--	--			
4-Chloroaniline	106-47-8	8270C	0.34	RSL	0.1			
4-Chlorophenyl phenyl ether	7005-72-3	8270C	--	--	--			
4-Methylphenol (p-cresol)	8001-28-3	8270C	18	RSL	6.0			
4-Nitroaniline	100-01-6	8270C	3.4	RSL	1.1			
4-Nitrophenol	824-78-2	8270C	--	--	--			
Acetophenone	98-86-2	8270C	370	RSL	123.3			
Atrazine	1912-24-9	8270C	3	MCL	1.0			
Benzaldehyde	100-52-7	8270C	370	RSL	123.3			
Bis(2-chloroethoxy)methane	111-91-1	8270C	11	RSL	3.7			
Bis(2-chloroethyl)ether	111-44-4	8270C	0.012	RSL	0.004			
Bis(2-ethylhexyl)phthalate	117-81-7	8270C	6	MCL	2.0			
Butyl benzyl phthalate	85-68-7	8270C	35	RSL	11.7			
Caprolactam	105-60-2	8270C	1800	RSL	600.0			
Carbazole	86-74-8	8270C	--	--	--			
Dibenzofuran	132-64-9	8270C	--	--	--			
Diethyl phthalate	84-66-2	8270C	2900	RSL	966.7			
Dimethyl phthalate	131-11-3	8270C	--	--	--			
Di-n-butyl phthalate	84-74-2	8270C	--	--	--			
Di-n-octyl phthalate	117-84-0	8270C	--	--	--			
Hexachlorobenzene	118-74-1	8270C SIM	1	MCL	0.3			
Hexachlorobutadiene	87-68-3	8270C	0.86	RSL	0.3			
Hexachlorocyclopentadiene	77-47-4	8270C	50	MCL	16.7			
Hexachloroethane	67-72-1	8270C	4.8	RSL	1.6			
Indeno(1,2,3-c,d)pyrene	193-39-5	8270C	--	--	--			
Isophorone	78-59-1	8270C	71	RSL	23.7			
Nitrobenzene	98-95-3	8270C	0.12	RSL	0.04			
N-Nitrosodi-n-propylamine	621-64-7	8270C	0.0096	RSL	0.003			
N-Nitrosodiphenylamine	86-30-6	8270C	14	RSL	4.7			
Pentachlorophenol	87-86-5	8270C SIM	1	MCL	0.3			
Phenol	108-95-2	8270C	1100	RSL	366.7			
PAHs (ug/L)								
2-Methylnaphthalene	91-57-6	8270C SIM	--	--	--			
Acenaphthene	83-32-9	8270C SIM	220	RSL	73.3			
Acenaphthylene	208-96-8	8270C SIM	--	--	--			
Anthracene	120-12-7	8270C SIM	1100	RSL	366.7			
Benzo(a)anthracene	56-55-3	8270C SIM	0.029	RSL	0.01			
Benzo(a)pyrene	50-32-8	8270C SIM	0.2	MCL	0.1			
Benzo(b)fluoranthene	205-99-2	8270C SIM	0.029	RSL	0.01			
Benzo(g,h,i)perylene	191-24-2	8270C SIM	--	--	--			
Benzo(k)fluoranthene	207-08-9	8270C SIM	0.29	RSL	0.1			
Chrysene	218-01-9	8270C SIM	2.9	RSL	1.0			
Dibenzo(a,h)anthracene	56-56-4	8270C SIM	0.0029	RSL	0.001			
Fluoranthene	206-44-0	8270C SIM	150	RSL	50.0			
Fluorene	86-73-7	8270C SIM	150	RSL	50.0			
Naphthalene	91-20-3	8270C SIM	0.14	RSL	0.05			
Phenanthrene	94-09-7	8270C SIM	--	--	--			
Pyrene	129-00-0	8270C SIM	110	RSL	36.7			

Pesticides (ug/L)								
4,4'-DDE	72-55-9	8081A	0.2	RSL	0.1			
4,4'-DDD	72-54-8	8081A	0.28	RSL	0.1			
4,4'-DDT	50-29-3	8081A	0.2	RSL	0.1			
Aldrin	309-00-2	8081A	0.004	RSL	0.001			
alpha-BHC	319-84-6	8081A	0.11	RSL	0.04			
alpha-Chlordane	5103-71-6	8081A	2	MCL	0.7			
beta-BHC	319-85-7	8081A	0.037	RSL	0.01			
delta-BHC	58-89-9	8081A	--	--	--			
Dieldrin	60-57-1	8081A	0.0042	RSL	0.001			
Endosulfan I	959-98-8	8081A	22	RSL	7.3			
Endosulfan II	33213-65-9	8081A	22	RSL	7.3			
Endosulfan sulfate	1031-07-8	8081A	--	--	--			
Endrin	72-20-8	8081A	2	MCL	0.7			
Endrin aldehyde	7421-93-4	8081A	--	--	--			
Endrin ketone	53494-70-5	8081A	--	--	--			
gamma-BHC (Lindane)	58-89-9	8081A	0.2	RSL	0.1			
gamma-Chlordane	5103-74-2	8081A	2	MCL	0.7			
Heptachlor	76-44-8	8081A	0.4	MCL	0.1			
Heptachlor epoxide	1024-57-3	8081A	0.2	MCL	0.1			
Methoxychlor	72-43-5	8081A	40	MCL	13.3			
Toxaphene	8001-35-2	8081A	3	MCL	1.0			
PCBs (ug/L)								
Aroclor-1016	12674-11-2	8082	0.5	MCL	0.2			
Aroclor-1221	11104-28-2	8082	0.5	MCL	0.2			
Aroclor-1232	11141-16-5	8082	0.5	MCL	0.2			
Aroclor-1242	53469-21-9	8082	0.5	MCL	0.2			
Aroclor-1248	12672-29-6	8082	0.5	MCL	0.2			
Aroclor-1254	11097-69-1	8082	0.5	MCL	0.2			
Aroclor-1260	11096-82-5	8082	0.5	MCL	0.2			
Total Polychlorinated Biphenyls	1336-36-3	--	0.5	MCL	0.2			
GRO and DRO (ug/L)								
GRO (C5-C12)	--	8015B	--	--	--			
DRO (C9-C36)	--	8015B	--	--	--			
Metals (Total and Dissolved) (ug/L)								
Aluminum	7429-90-5	6010B	3700	RSL	1233.3			
Antimony	7440-36-0	6010B	6	MCL	2.0			
Arsenic	7440-38-2	6010B	10	MCL	3.3			
Barium	7440-39-3	6010B	2000	MCL	666.7			
Beryllium	7440-41-7	6010B	4	MCL	1.3			
Cadmium	7440-43-9	6010B	5	MCL	1.7			
Calcium	7440-70-2	6010B	--	--	--			
Chromium	7440-47-3	6010B	100	MCL	33.3			
Cobalt	7440-48-4	6010B	1.1	RSL	0.4			
Copper	7440-50-8	6010B	1300	MCL	433.3			
Iron	7439-89-6	6010B	2600	RSL	866.7			
Lead	7439-92-1	6010B	15	MCL	5.0			
Magnesium	7439-95-4	6010B	--	--	--			
Manganese	7439-96-5	6010B	88	RSL	29.3			
Mercury	7439-97-6	6010B	2	RIDEM GA Aquifer	0.7			
Nickel	7440-02-0	6010B	100	RIDEM GA Aquifer	33.3			
Potassium	7440-09-7	6010B	--	--	--			
Selenium	7782-49-2	6010B	50	MCL	16.7			
Silver	7440-22-4	6010B	18	RSL	6.0			
Sodium	7440-23-5	6010B	--	--	--			
Thallium	7440-28-0	6010B	2	MCL	0.7			
Vanadium	7440-62-2	6010B	0.26	RSL	0.1			
Zinc	7440-66-6	6010B	1100	RSL	366.7			

-- - Not Available

1. See units for each analyte group in first column. If no PQL Goal is available, or if laboratory cannot achieve the PQL Goal, laboratory should enter its lowest achievable QL and corresponding LOD and MDL for the method indicated

ATTACHMENT A
SOIL REQUIRED TARGET COMPOUND LIST AND PROJECT QUANTITATION LIMIT GOALS
SITE 4, CODDINGTON COVE RUBBLE FILL AREA, NAVSTA NEWPORT, NEWPORT, RI

Soil Samples - Analyte	CAS Number	Analytical Method	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Laboratory-specific[1]		
						LOQ	LOD	MDL
VOCs (mg/kg)								
1,1,1-Trichloroethane	71-55-6	8260B	0.07	TV	0.02			
1,1,2,2-Tetrachloroethane	79-34-5	8260B	0.56	RSL	0.2			
1,1,2-Trichloroethane	79-00-5	8260B	0.4	TV	0.1			
1,1-Dichloroethane	75-34-3	8260B	0.02	TV	0.01			
1,1-Dichloroethene	75-35-4	8260B	0.1	TV	0.03			
1,2,4-Trichlorobenzene	87-61-6	8260B	20	ORNL	6.7			
1,2-Dibromoethane (EDB)	80-97-7	8260B	0.034	RSL	0.01			
1,2-Dibromo-3-Chloropropane (DBCP)	96-12-8	8260B	0.0054	RSL	0.002			
1,2-Dichlorobenzene	95-50-1	8260B	0.03	TV	0.01			
1,2-Dichloroethane	107-06-2	8260B	0.02	TV	0.01			
1,2-Dichloropropane	78-87-5	8260B	0.89	RSL	0.3			
1,3-Dichlorobenzene	541-73-1	8260B	0.03	TV	0.01			
1,4-Dichlorobenzene	92-87-5	8260B	2.4	RSL	0.8			
2-Butanone	78-93-3	8260B	2800	RSL	933.3			
2-Hexanone	591-78-6	8260B	21	RSL	7.0			
4-Methyl-2-pentanone	108-10-1	8260B	58	RSL	19.3			
Acetone	67-64-1	8260B	6,100	RSL	2033			
Benzene	71-43-2	8260B	1.1	RSL	0.4			
Bromodichloromethane	75-27-4	8260B	0.27	RSL	0.1			
Bromoform	75-25-2	8260B	61	RSL	20.3			
Bromoethane	74-96-4	8260B	0.73	RSL	0.2			
Carbon disulfide	75-15-0	8260B	82	RSL	27.3			
Carbon tetrachloride	56-23-5	8260B	0.25	RSL	0.1			
Chlorobenzene	108-90-7	8260B	29	RSL	9.7			
Chloroethane	75-00-3	8260B	1500	RSL	500.0			
Chloroform	67-66-3	8260B	0.02	TV	0.01			
Chloromethane	74-87-3	8260B	12	RSL	4.0			
cis-1,2-Dichloroethene	156-59-2	8260B	0.2	TV	0.1			
cis-1,3-Dichloropropene	10061-01-5	8260B	1.7	RSL	0.6			
Cyclohexane	110-82-7	8260B	700	RSL	233.3			
Dibromochloromethane	124-48-1	8260B	0.68	RSL	0.2			
Dichlorodifluoromethane	75-71-8	8260B	18	RSL	6.0			
Ethylbenzene	100-41-4	8260B	1.2	CCME	0.4			
Isopropylbenzene	98-82-8	8260B	2100	RSL	700.0			
Methyl acetate	79-20-9	8260B	7800	RSL	2600			
Methylcyclohexane	108-87-2	8260B	--	--	--			
Methylene chloride	75-09-2	8260B	0.4	TV	0.1			
Methyl-tert-butyl ether	1634-04-4	8260B	43	RSL	14.3			
Styrene	100-42-5	8260B	300	ORNL	100.0			
Tetrachloroethene	127-18-4	8260B	0.55	RSL	0.2			
Toluene	108-88-3	8260B	200	ORNL	66.7			
trans-1,2-Dichloroethene	540-59-0	8260B	15	RSL	5.0			
trans-1,3-Dichloropropene	542-75-6	8260B	1.7	RSL	0.6			
Trichloroethene	86-42-0	8260B	2.8	RSL	0.9			
Trichlorofluoromethane	75-69-4	8260B	79	RSL	26.3			
Vinyl chloride	75-01-4	8260B	0.01	TV	0.003			
Xylenes (total)	1330-20-7	8260B	1	CCME	0.3			
PAHs (mg/kg)								
2-Methylnaphthalene	91-57-6	8270C SIM	3.6	RSL	1.2			
Acenaphthene	83-32-9	8270C SIM	29	Eco SSL	9.7			
Acenaphthylene	208-96-8	8270C SIM	3.6	RSL	1.2			
Anthracene	120-12-7	8270C SIM	29	Eco SSL	9.7			
Benzo(a)anthracene	56-55-3	8270C SIM	0.15	RSL	0.1			
Benzo(a)pyrene	50-32-8	8270C SIM	0.015	RSL	0.01			
Benzo(b)fluoranthene	205-99-2	8270C SIM	0.15	RSL	0.1			
Benzo(g,h,i)perylene	191-24-2	8270C SIM	1.1	Eco SSL	0.4			
Benzo(k)fluoranthene	207-08-9	8270C SIM	1.1	Eco SSL	0.4			
Chrysene	218-01-9	8270C SIM	1.1	Eco SSL	0.4			
Dibenzo(a,h)anthracene	56-56-4	8270C SIM	0.015	RSL	0.01			
Fluoranthene	206-44-0	8270C SIM	29	Eco SSL	9.7			
Fluorene	86-73-7	8270C SIM	29	Eco SSL	9.7			
Naphthalene	91-20-3	8270C SIM	3.6	RSL	1.2			
Phenanthrene	94-09-7	8270C SIM	29	Eco SSL	9.7			
Pyrene	129-00-0	8270C SIM	1.1	Eco SSL	0.4			

Pesticides (mg/kg)								
4,4'-DDE	72-55-9	8081A	2	RSL	0.7			
4,4'-DDD	72-54-8	8081A	1.4	RSL	0.5			
4,4'-DDT	50-29-3	8081A	0.021	Eco SSL	0.01			
Aldrin	309-00-2	8081A	0.00006	TV	0.00002			
alpha-BHC	319-84-6	8081A	0.003	TV	0.001			
alpha-Chlordane	5103-71-6	8081A	0.00003	TV	0.00001			
beta-BHC	319-85-7	8081A	0.009	TV	0.003			
delta-BHC	58-89-9	8081A	0.01	TV	0.003			
Dieldrin	60-57-1	8081A	0.0049	Eco SSL	0.002			
Endosulfan I	959-98-8	8081A	0.00001	TV	0.000003			
Endosulfan II	33213-65-9	8081A	0.00001	TV	0.000003			
Endosulfan sulfate	1031-07-8	8081A	0.00001	TV	0.000003			
Endrin	72-20-8	8081A	0.00004	TV	0.00001			
Endrin aldehyde	7421-93-4	8081A	0.00004	TV	0.00001			
Endrin ketone	53494-70-5	8081A	0.00004	TV	0.00001			
gamma-BHC (Lindane)	58-89-9	8081A	0.00005	TV	0.00002			
gamma-Chlordane	5103-74-2	8081A	0.00003	TV	0.00001			
Heptachlor	76-44-8	8081A	0.0007	TV	0.0002			
Heptachlor epoxide	1024-57-3	8081A	0.0000002	TV	0.0000001			
Methoxychlor	72-43-5	8081A	31	RSL	10.3			
Toxaphene	8001-35-2	8081A	0.44	RSL	0.1			
PCBs (mg/kg)								
Aroclor-1016	12674-11-2	8082	3.9	RSL	1.3			
Aroclor-1221	11104-28-2	8082	0.14	RSL	0.05			
Aroclor-1232	11141-16-5	8082	0.14	RSL	0.05			
Aroclor-1242	53469-21-9	8082	0.22	RSL	0.1			
Aroclor-1248	12672-29-6	8082	0.22	RSL	0.1			
Aroclor-1254	11097-69-1	8082	0.22	RSL	0.1			
Aroclor-1260	11096-82-5	8082	0.22	RSL	0.1			
Total Polychlorinated Biphenyls	1336-36-3		--	--	1.3			
GRO and DRO (mg/kg)								
GRO (C5-C12)		8015B	500	RIDEM TPH	166.7			
DRO (C9-C36)		8015B	500	RIDEM TPH	166.7			
Metals (mg/kg)								
Aluminum	7429-90-5	6010B	7700	RSL	2567			
Antimony	7440-36-0	6010B	0.27	Eco SSL	0.1			
Arsenic	7440-38-2	6010B	0.39	RSL	0.1			
Barium	7440-39-3	6010B	330	Eco SSL	110.0			
Beryllium	7440-41-7	6010B	16	RSL	5.3			
Cadmium	7440-43-9	6010B	0.36	Eco SSL	0.1			
Calcium	7440-70-2	6010B	--	--	--			
Chromium	7440-47-3	6010B	0.29	RSL	0.1			
Cobalt	7440-48-4	6010B	2.3	RSL	0.8			
Copper	7440-50-8	6010B	28	Eco SSL	9.3			
Iron	7439-89-6	6010B	5500	RSL	1833			
Lead	7439-92-1	6010B	11	Eco SSL	3.7			
Magnesium	7439-95-4	6010B	--	--	--			
Manganese	7439-96-5	6010B	180	RSL	60.0			
Mercury	7439-97-6	6010B	0.00051	ORNL	0.00			
Nickel	7440-02-0	6010B	38	Eco SSL	12.7			
Potassium	7440-09-7	6010B	--	--	--			
Selenium	7782-49-2	6010B	0.52	Eco SSL	0.2			
Silver	7440-22-4	6010B	4.2	Eco SSL	1.4			
Sodium	7440-23-5	6010B	--	--	--			
Thallium	7440-28-0	6010B	1	ORNL	0.3			
Vanadium	7440-62-2	6010B	0.55	RSL	0.2			
Zinc	7440-66-6	6010B	46	Eco SSL	15.3			

-- - Not Available

1. See units for each analyte group in first column. If no PQL Goal is available, or if laboratory cannot achieve the PQL Goal, laboratory should enter its lowest achievable QL and corresponding LOD and MDL for the method indicated

ATTACHMENT A
SEDIMENT REQUIRED TARGET COMPOUND LIST AND PROJECT QUANTITATION LIMIT GOALS
SITE 4, CODDINGTON COVE RUBBLE FILL AREA, NAVSTA NEWPORT, NEWPORT, R

Sediment Samples - Analyte	CAS Number	Analytical Method	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Laboratory-specific[1]		
						LOQ	LOD	MDL
VOCs (ug/kg)								
1,1,1-Trichloroethane	71-55-6	8260B	170	EPA SQB	56.7			
1,1,2,2-Tetrachloroethane	79-34-5	8260B	940	EPA SQB	313.3			
1,1,2-Trichloroethane	79-00-5	8260B	1200	SCV	400.0			
1,1-Dichloroethane	75-34-3	8260B	27	SCV	9.0			
1,1-Dichloroethene	75-35-4	8260B	31	SCV	10.3			
1,2,4-Trichlorobenzene	87-61-6	8260B	9200	EPA SQB	3067			
1,2-Dibromoethane (EDB)	80-97-7	8260B	--	--	--			
1,2-Dibromo-3-Chloropropane (DBCP)	96-12-8	8260B	--	--	--			
1,2-Dichlorobenzene	95-50-1	8260B	340	EPA SQB	113.3			
1,2-Dichloroethane	107-06-2	8260B	250	SCV	83.3			
1,2-Dichloropropane	78-87-5	8260B	--	--	--			
1,3-Dichlorobenzene	541-73-1	8260B	1700	EPA SQB	566.7			
1,4-Dichlorobenzene	92-87-5	8260B	350	EPA SQB	116.7			
2-Butanone	78-93-3	8260B	270	SCV	90.0			
2-Hexanone	591-78-6	8260B	22	SCV	7.3			
4-Methyl-2-pentanone	108-10-1	8260B	33	SCV	11.0			
Acetone	67-64-1	8260B	9	SCV	3.0			
Benzene	71-43-2	8260B	57	EPA SQB	19.0			
Bromodichloromethane	75-27-4	8260B	--	--	--			
Bromoform	75-25-2	8260B	650	EPA SQB	216.7			
Bromoethane	74-96-4	8260B	--	--	--			
Carbon disulfide	75-15-0	8260B	0.85	SCV	0.3			
Carbon tetrachloride	56-23-5	8260B	1200	EPA SQB	400.0			
Chlorobenzene	108-90-7	8260B	820	EPA SQB	273.3			
Chloroethane	75-00-3	8260B	--	--	--			
Chloroform	67-66-3	8260B	22	SCV	7.3			
Chloromethane	74-87-3	8260B	--	--	--			
cis-1,2-Dichloroethene	156-59-2	8260B	400	SCV	133.3			
cis-1,3-Dichloropropene	10061-01-5	8260B	0.051	SCV	0.02			
Cyclohexane	110-82-7	8260B	--	--	--			
Dibromochloromethane	124-48-1	8260B	--	--	--			
Dichlorodifluoromethane	75-71-8	8260B	--	--	--			
Ethylbenzene	100-41-4	8260B	3600	EPA SQB	1200			
Isopropylbenzene	98-82-8	8260B	--	--	--			
Methyl acetate	79-20-9	8260B	--	--	--			
Methylcyclohexane	108-87-2	8260B	--	--	--			
Methylene chloride	75-09-2	8260B	370	SCV	123.3			
Methyl-tert-butyl ether	1634-04-4	8260B	--	--	--			
Styrene	100-42-5	8260B	--	--	--			
Tetrachloroethene	127-18-4	8260B	530	EPA SQB	176.7			
Toluene	108-88-3	8260B	670	EPA SQB	223.3			
trans-1,2-Dichloroethene	540-59-0	8260B	400	SCV	133.3			
trans-1,3-Dichloropropene	542-75-6	8260B	0.051	SCV	0.02			
Trichloroethene	86-42-0	8260B	1600	EPA SQB	533.3			
Trichlorofluoromethane	75-69-4	8260B	--	--	--			
Vinyl chloride	75-01-4	8260B	--	--	--			
Xylenes (total)	1330-20-7	8260B	130	SCV	43.3			
PAHs (ug/kg)								
2-Methylnaphthalene	91-57-6	8270C SIM	20.2	ESV	6.7			
Acenaphthene	83-32-9	8270C SIM	290	NOAA	96.7			
Acenaphthylene	208-96-8	8270C SIM	160	NOAA	53.3			
Anthracene	120-12-7	8270C SIM	57.2	TEC	19.1			
Benzo(a)anthracene	56-55-3	8270C SIM	108	TEC	36.0			
Benzo(a)pyrene	50-32-8	8270C SIM	150	TEC	50.0			
Benzo(b)fluoranthene	205-99-2	8270C SIM	1800	NOAA	600.0			
Benzo(g,h,i)perylene	191-24-2	8270C SIM	170	LEL	56.7			
Benzo(k)fluoranthene	207-08-9	8270C SIM	240	LEL	80.0			
Chrysene	218-01-9	8270C SIM	166	TEC	55.3			
Dibenzo(a,h)anthracene	56-56-4	8270C SIM	33	TEC	11.0			
Fluoranthene	206-44-0	8270C SIM	423	TEC	141.0			
Fluorene	86-73-7	8270C SIM	77.4	TEC	25.8			
Naphthalene	91-20-3	8270C SIM	176	TEC	58.7			
Phenanthrene	94-09-7	8270C SIM	204	TEC	68.0			
Pyrene	129-00-0	8270C SIM	195	TEC	65.0			

Pesticides (ug/kg)								
4,4'-DDE	72-55-9	8081A	3.16	TEC	1.1			
4,4'-DDD	72-54-8	8081A	4.88	TEC	1.6			
4,4'-DDT	50-29-3	8081A	4.16	TEC	1.4			
Aldrin	309-00-2	8081A	2	LEL	0.7			
alpha-BHC	319-84-6	8081A	6	LEL	2.0			
alpha-Chlordane	5103-71-6	8081A	3.24	TEC	1.1			
beta-BHC	319-85-7	8081A	5	LEL	1.7			
delta-BHC	58-89-9	8081A	120	SCV	40.0			
Dieldrin	60-57-1	8081A	2	LEL	0.7			
Endosulfan I	959-98-8	8081A	2.9	SQB	1.0			
Endosulfan II	33213-65-9	8081A	14	SQB	4.7			
Endosulfan sulfate	1031-07-8	8081A	5.4	SQB	1.8			
Endrin	72-20-8	8081A	2	TEC	0.7			
Endrin aldehyde	7421-93-4	8081A	--	--	--			
Endrin ketone	53494-70-5	8081A	--	--	--			
gamma-BHC (Lindane)	58-89-9	8081A	2.37	TEC	0.8			
gamma-Chlordane	5103-74-2	8081A	3.24	TEC	1.1			
Heptachlor	76-44-8	8081A	10	NOAA	3.3			
Heptachlor epoxide	1024-57-3	8081A	2.47	TEC	0.8			
Methoxychlor	72-43-5	8081A	19	SQB	6.3			
Toxaphene	8001-35-2	8081A	28	SQB	9.3			
PCBs (ug/kg)								
Aroclor-1016	12674-11-2	8082	59.8	TEC	19.9			
Aroclor-1221	11104-28-2	8082	59.8	TEC	19.9			
Aroclor-1232	11141-16-5	8082	59.8	TEC	19.9			
Aroclor-1242	53469-21-9	8082	59.8	TEC	19.9			
Aroclor-1248	12672-29-6	8082	59.8	TEC	19.9			
Aroclor-1254	11097-69-1	8082	59.8	TEC	19.9			
Aroclor-1260	11096-82-5	8082	59.8	TEC	19.9			
Total Polychlorinated Biphenyls	1336-36-3		59.8	TEC	19.9			
GRO and DRO (mg/kg)								
GRO (C5-C12)		8015B	500	RIDEM TPH	166.7			
DRO (C9-C36)		8015B	500	RIDEM TPH	166.7			
Metals (mg/kg)								
Aluminum	7429-90-5	6010B	--	--	--			
Antimony	7440-36-0	6010B	3	NOAA	1.0			
Arsenic	7440-38-2	6010B	9.79	TEC	3.3			
Barium	7440-39-3	6010B	48	NOAA	16.0			
Beryllium	7440-41-7	6010B	--	--	--			
Cadmium	7440-43-9	6010B	0.99	TEC	0.3			
Calcium	7440-70-2	6010B	--	--	--			
Chromium	7440-47-3	6010B	43.4	TEC	14.5			
Cobalt	7440-48-4	6010B	50	NOAA	16.7			
Copper	7440-50-8	6010B	31.6	TEC	10.5			
Iron	7439-89-6	6010B	20000	LEL	6667			
Lead	7439-92-1	6010B	35.8	TEC	11.9			
Magnesium	7439-95-4	6010B	--	--	--			
Manganese	7439-96-5	6010B	460	LEL	153.3			
Mercury	7439-97-6	6010B	0.18	TEC	0.1			
Nickel	7440-02-0	6010B	22.7	TEC	7.6			
Potassium	7440-09-7	6010B	--	--	--			
Selenium	7782-49-2	6010B	1	NOAA	0.3			
Silver	7440-22-4	6010B	0.5	LEL	0.2			
Sodium	7440-23-5	6010B	--	--	--			
Thallium	7440-28-0	6010B	--	--	--			
Vanadium	7440-62-2	6010B	57	NOAA	19.0			
Zinc	7440-66-6	6010B	121	TEC	40.3			

-- - Not available or not applicable

1. See units for each analyte group in first column. If no PQL Goal is available, or if laboratory cannot achieve the PQL Goal, laboratory should enter its lowest achievable QL and corresponding LOD and MDL for the method indicated.

ATTACHMENT A
SURFACE WATER REQUIRED TARGET COMPOUND LIST AND PROJECT QUANTITATION LIMIT GOALS
SITE 4, CODDINGTON COVE RUBBLE FILL AREA, NAVSTA NEWPORT, NEWPORT, RI

Surface Water Samples - Analyte	CAS Number	Analytical Method	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Laboratory-specific[1]		
						LOQ	LOD	MDL
VOCs (ug/L)								
1,1,1-Trichloroethane	71-55-6	8260B	11	SCV	3.7			
1,1,2,2-Tetrachloroethane	79-34-5	8260B	10	RIDEM	3.3			
1,1,2-Trichloroethane	79-00-5	8260B	20	RIDEM	6.7			
1,1-Dichloroethane	75-34-3	8260B	47	SCV	15.7			
1,1-Dichloroethene	75-35-4	8260B	13	RIDEM	4.3			
1,2,4-Trichlorobenzene	87-61-6	8260B	1.7	RIDEM	0.6			
1,2-Dibromoethane (EDB)	80-97-7	8260B	--	--	--			
1,2-Dibromo-3-Chloropropane (DBCP)	96-12-8	8260B	--	--	--			
1,2-Dichlorobenzene	95-50-1	8260B	1.8	RIDEM	0.6			
1,2-Dichloroethane	107-06-2	8260B	131	RIDEM	43.7			
1,2-Dichloropropane	78-87-5	8260B	58	RIDEM	19.3			
1,3-Dichlorobenzene	541-73-1	8260B	8.7	RIDEM	2.9			
1,4-Dichlorobenzene	92-87-5	8260B	1.2	RIDEM	0.4			
2-Butanone	78-93-3	8260B	14000	SCV	4667			
2-Hexanone	591-78-6	8260B	99	SCV	33.0			
4-Methyl-2-pentanone	108-10-1	8260B	170	SCV	56.7			
Acetone	67-64-1	8260B	1500	SCV	500.0			
Benzene	71-43-2	8260B	5.9	RIDEM	2.0			
Bromodichloromethane	75-27-4	8260B	11000	NOAA	3667			
Bromoform	75-25-2	8260B	33	RIDEM	11.0			
Bromoethane	74-96-4	8260B	--	--	--			
Carbon disulfide	75-15-0	8260B	0.92	SCV	0.3			
Carbon tetrachloride	56-23-5	8260B	30	RIDEM	10.0			
Chlorobenzene	108-90-7	8260B	18	RIDEM	6.0			
Chloroethane	75-00-3	8260B	--	--	--			
Chloroform	67-66-3	8260B	32	RIDEM	10.7			
Chloromethane	74-87-3	8260B	--	--	--			
cis-1,2-Dichloroethene	156-59-2	8260B	590	SCV	196.7			
cis-1,3-Dichloropropene	10061-01-5	8260B	0.055	SCV	0.02			
Cyclohexane	110-82-7	8260B	--	--	--			
Dibromochloromethane	124-48-1	8260B	11000	NOAA	3667			
Dichlorodifluoromethane	75-71-8	8260B	--		--			
Ethylbenzene	100-41-4	8260B	36	RIDEM	12.0			
Isopropylbenzene	98-82-8	8260B	--	--	--			
Methyl acetate	79-20-9	8260B	--	--	--			
Methylcyclohexane	108-87-2	8260B	--	--	--			
Methylene chloride	75-09-2	8260B	214	RIDEM	71.3			
Methyl-tert-butyl ether	1634-04-4	8260B	10000	NOAA	3333			
Styrene	100-42-5	8260B	72	NOAA	24.0			
Tetrachloroethene	127-18-4	8260B	5.3	RIDEM	1.8			
Toluene	108-88-3	8260B	14	RIDEM	4.7			
trans-1,2-Dichloroethene	540-59-0	8260B	590	SCV	196.7			
trans-1,3-Dichloropropene	542-75-6	8260B	0.055	SCV	0.02			
Trichloroethene	86-42-0	8260B	43	RIDEM	14.3			
Trichlorofluoromethane	75-69-4	8260B	11000	NOAA	3667			
Vinyl chloride	75-01-4	8260B	930	NOAA	310.0			
Xylenes (total)	1330-20-7	8260B	3	RIDEM	1.0			
PAHs (ug/L)								
2-Methylnaphthalene	91-57-6	8270C SIM	30	NOAA	10.0			
Acenaphthene	83-32-9	8270C SIM	1	RIDEM	0.3			
Acenaphthylene	208-96-8	8270C SIM	484	NOAA	161.3			
Anthracene	120-12-7	8270C SIM	0.73	SCV	0.2			
Benzo(a)anthracene	56-55-3	8270C SIM	0.027	SCV	0.01			
Benzo(a)pyrene	50-32-8	8270C SIM	0.014	SCV	0.005			
Benzo(b)fluoranthene	205-99-2	8270C SIM	0.9	NOAA	0.3			
Benzo(g,h,i)perylene	191-24-2	8270C SIM	0.76	NOAA	0.3			
Benzo(k)fluoranthene	207-08-9	8270C SIM	0.6415	4-Mar	0.2			
Chrysene	218-01-9	8270C SIM	2.042	4-Mar	0.7			
Dibenzo(a,h)anthracene	56-56-4	8270C SIM	0.2825	4-Mar	0.1			
Fluoranthene	206-44-0	8270C SIM	4.4	NOAA	1.5			
Fluorene	86-73-7	8270C SIM	3.9	SCV	1.3			
Naphthalene	91-20-3	8270C SIM	2.6	RIDEM	0.9			
Phenanthrene	94-09-7	8270C SIM	6.3	SCV	2.1			
Pyrene	129-00-0	8270C SIM	0.025	NOAA	0.01			
Pesticides (ug/L)								
4,4'-DDE	72-55-9	8081A	105	NOAA	35.0			
4,4'-DDD	72-54-8	8081A	0.011	SCV	0.004			
4,4'-DDT	50-29-3	8081A	0.001	RIDEM	0.0003			
Aldrin	309-00-2	8081A	3	NRWQC	1.0			
alpha-BHC	319-84-6	8081A	2.2	SCV	0.7			
alpha-Chlordane	5103-71-6	8081A	0.0043	NRWQC	0.0			
beta-BHC	319-85-7	8081A	2.2	SCV	0.7			
delta-BHC	58-89-9	8081A	2.2	SCV	0.7			
Dieldrin	60-57-1	8081A	0.056	NRWQC	0.02			
Endosulfan I	959-98-8	8081A	0.056	NRWQC	0.02			
Endosulfan II	33213-65-9	8081A	0.056	NRWQC	0.02			
Endosulfan sulfate	1031-07-8	8081A	0.051	SCV	0.02			
Endrin	72-20-8	8081A	0.036	NRWQC	0.01			
Endrin aldehyde	7421-93-4	8081A	0.15	NOAA	0.1			

Endrin ketone	53494-70-5	8081A	--	--	--			
gamma-BHC (Lindane)	58-89-9	8081A	0.08	NOAA	0.03			
gamma-Chlordane	5103-74-2	8081A	0.0043	NRWQC	0.001			
Heptachlor	76-44-8	8081A	0.0038	NRWQC	0.001			
Heptachlor epoxide	1024-57-3	8081A	0.0038	NRWQC	0.001			
Methoxychlor	72-43-5	8081A	0.019	SCV	0.006			
Toxaphene	8001-35-2	8081A	0.0002	NRWQC	0.000			
PCBs (ug/L)								
Aroclor-1016	12674-11-2	8082	0.014	NRWQC	0.005			
Aroclor-1221	11104-28-2	8082	0.014	NRWQC	0.005			
Aroclor-1232	11141-16-5	8082	0.014	NRWQC	0.005			
Aroclor-1242	53469-21-9	8082	0.014	NRWQC	0.005			
Aroclor-1248	12672-29-6	8082	0.014	NRWQC	0.005			
Aroclor-1254	11097-69-1	8082	0.014	NRWQC	0.005			
Aroclor-1260	11096-82-5	8082	0.014	NRWQC	0.005			
Total Polychlorinated Biphenyls	1336-36-3		0.014	NRWQC	0.005			
GRO and DRO (ug/L)								
GRO (C5-C12)		8015B	--		--			
DRO (C9-C36)		8015B	--		--			
Metals (ug/L)								
Aluminum	7429-90-5	6010B	87	NRWQC	29.0			
Antimony	7440-36-0	6010B	10	RIDEM	3.3			
Arsenic	7440-38-2	6010B	150	NRWQC	50.0			
Barium	7440-39-3	6010B	4	SCV	1.3			
Beryllium	7440-41-7	6010B	0.17	RIDEM	0.06			
Cadmium	7440-43-9	6010B	0.25	NRWQC	0.08			
Calcium	7440-70-2	6010B	--	--	--			
Chromium	7440-47-3	6010B	11	NRWQC	3.7			
Cobalt	7440-48-4	6010B	23	SCV	7.7			
Copper	7440-50-8	6010B	9	NRWQC	3.0			
Iron	7439-89-6	6010B	1000	NRWQC	333.3			
Lead	7439-92-1	6010B	2.5	NRWQC	0.8			
Magnesium	7439-95-4	6010B	--	--	--			
Manganese	7439-96-5	6010B	120	SCV	40.0			
Mercury	7439-97-6	6010B	0.77	NRWQC	0.3			
Nickel	7440-02-0	6010B	52	NRWQC	17.3			
Potassium	7440-09-7	6010B	--	--	--			
Selenium	7782-49-2	6010B	5	NRWQC	1.7			
Silver	7440-22-4	6010B	0.36	SCV	0.1			
Sodium	7440-23-5	6010B	--	--	--			
Thallium	7440-28-0	6010B	1	RIDEM	0.3			
Vanadium	7440-62-2	6010B	20	SCV	6.7			
Zinc	7440-66-6	6010B	120	NRWQC	40.0			

-- - Not Available

1. See units for each analyte group in first column. If no PQL Goal is available, or if laboratory cannot achieve the PQL Goal, laboratory should enter its lowest achievable QL and corresponding LOD and MDL for the method indicated

ATTACHMENT B
DATA PACKAGE DELIVERABLES REQUIREMENTS

DATA PACKAGE DELIVERABLE REQUIREMENTS

The laboratory is to provide a hard copy plus two compact disks (CDs) each containing a PDF file in the following format:

1. Table of Contents
2. Case Narrative
3. Chain-of-Custody
4. Data Summary Package (contains summary of all CLP or CLP-like Forms 1 through 15 per analytical fraction)
5. Analytical Fractions (VOA, SVOC, Metals, etc.)
    a. Results and QC Summary (summary of all CLP or CLP like Forms 1 through 15 for a particular analytical fraction)
    b. Raw Sample Data (includes all sample dilutions, sample re-analyses, QC samples, etc.)
    c. Calibration Data (includes all initial and continuing calibrations)
    d. Miscellaneous (includes extraction/preparation forms, percent solids determination, IDLs, MDLs, etc.)

Each of the above sections should be bookmarked in the PDF for easy access.

Summary Form Requirements for PDF data package deliverable for non-CLP Methods:

The following summary forms are required as part of the data package deliverable for SW-846 6020/6010B/7000A series for metals:

Results Report - must present all information presented on CLP FORM I.
Initial and Continuing Calibration Summary -must present all information presented on CLP FORM 2A.
Blanks - must present all information presented on CLP FORM 3.
ICP Interference Check Sample Summary - must present all information presented on CLP FORM 4.
Matrix Spike Summary - must present all information presented on CLP FORM 5A.
Post Digestion Spike - must present all information presented on CLP FORM 5B.
Lab Duplicate Results - must present all information presented on CLP FORM 6.
LCS Summary - must present all information presented on CLP FORM 7.
MSA Summary (Method of Standard Addition) - must present all information presented on CLP FORM 8.
ICP Serial Dilution Summary - must present all information presented on CLP FORM 9.
Detection Limits - must present all information presented on CLP FORM 10.
Linear Range – must present all information presented on CLP FORM 12.
Prep Log - must present all information presented on CLP FORM 13.
Analysis Run Log - must present all information presented on CLP FORM 14.
ICP-MS Tune – must present all information presented on CLP FORM 14 (ILM05.4).
ICP/MS Internal Standard Relative Intensity Summary - must present all information presented on CLP FORM 15 (ILM05.4).

Also must include: Instrument Calibration Records, Chain-of-Custody Forms, and Case Narrative.

Summary Forms for SW-846, 8260B and 8270C (i.e., Any SW-846 GC/MS analysis of Volatile and Semivolatile Organic Compounds) should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Instrument Performance Check Summary Form - Mass Spec Tuning Form	Present all information Contained on CLP Form V.
Initial Calibration Summary	Present all information contained CLP Form VI.
Continuing Calibration Summary	Present All Information contained on CLP Form VII.
Internal Standard Area and Retention Time Summary	Present all information contained CLP Form VIII.

Summary Forms for SW-846 8081A and 8082 Pesticide and Polychlorinated Biphenyl (PCB) Organic Compounds (and 8151A, 8141A, and all other SW-846 GC methods) should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II for both Analytical Columns.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Summary of Pesticide Initial Calibration of Single Component Analytes	Present all information contained on CLP Form VI-PEST-2.
Summary of Pesticide Calibration Verification	Present all information contained on CLP Form VII-PEST-1 and Form VII-PEST-2.
Pesticide Analytical Sequence	Present all information contained on CLP Form VIII-PEST.
Pesticide Identification Summary For Single Component Analytes and for Multiple Component Analytes	Present all information contained on CLP Form X PEST 1 and 2.

ATTACHMENT C
ELECTRONIC DATA DELIVERABLE REQUIREMENTS

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to submit text-based tab delimited EDD files for each SDG using Tetra Tech's laboratory data checker explained below. The files must be in the format specified in this Attachment. Additional information such as laboratory name, project name, fractions included, project number, site name/number, laboratory contact person and any specific comments related to the EDD should be included in the comments section of the EDD Submittal page.

The RESULT for nondetects should be populated with the project-specific sample quantitation reporting limits (i.e., either the sample quantitation limit or method detection limit, as specified in Section 3.0 of this scope of work. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Tetra Tech's electronic EDD format follows the ADAPT structure and requires the A1 and A3 files. The A2 file is only required if the project is using ADAPT; and, for non-ADAPT EDD submittals the A2 file may be omitted. The EDD consists of separate, tab-delimited ASCII text files. Each file corresponds to a database table. The tables are identified as the Analytical Results Table (A1) and Sample Analysis Table (A3). A separate set of text files must be created and submitted for each sample delivery group (SDG). The files must be identified to correspond to the (A1) table and the (A3) table. The file naming convention is: the Sample Delivery Group (SDG) followed by the table identifier (A1 or A3), followed by the ".txt" extension. The file names must not contain spaces or special characters. For example, the EDD file names for a laboratory-reporting batch identified as SDG001 would be as follows:

SDG001A1.txt
SDG001A3.txt

On certain projects Tetra Tech will utilize the ADAPT Electronic Data Validation software, which will require the laboratory to use the ADAPT electronic data deliverable checker software prior to submitting the files through Tetra Tech's laboratory data checker (this will be clearly specified in the Tetra Tech laboratory statement of work). The ADAPT checker software can be downloaded from Laboratory Data Consultants' web site: http://www.lab-data.com. For projects which Tetra Tech is using the ADAPT software, Tetra Tech will provide the laboratory with the project library. The laboratory is not permitted to modify the project library. ADAPT projects will require the laboratory to export all three checked files (A1, A2, and A3) from the ADAPT software and submit them through Tetra Tech's laboratory data checker. **ADAPT error logs generated must be included with the electronic PDF data validation package and cannot be submitted through the laboratory data checker.**

The values reported in the EDD text files must agree exactly with the final values reported on the PDF data package sample result summaries. The details of file naming conventions, data structure and data checker use are discussed below.

Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information for target analytes in field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field samples and laboratory method blanks must report a result record for each analyte reported within a method. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the laboratory statement of work. Table A1 in this document lists the field names and data type descriptions for the Analytical Results Table (A1).

Lab Instrument Table (A2 File)

A2 file is only required if the project is using ADAPT. In all other EDD submittals, the A2 file may be omitted. Laboratories should refer to the ADAPT User Guide for populating the A2 Table.

Sample Analysis Table (A3 File)

The Sample Analysis table contains information specific to field environmental samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record must exist for each sample/method/matrix/analysis type combination. Table A3 in this document lists the field names and data type descriptions for the Sample Analysis Table (A3).

All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at
(____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____Date: _____

2.0 EDD Field Properties

Tables A1 and A3 in this document specify the EDD field properties. Laboratories should refer to the ADAPT User Guide for populating the A2 Table. These include the field name, sequence order, field description, data type/length and reporting requirement for each field. Fields in the EDD **must** be sequenced according to the order that they appear below in Tables A1 and A3. For example, in the Analytical Results table (A1), the field "ClientSampleID" will always be the first piece of information to start every new line of data (or database record), followed by the field "LabAnalysisRefMethodID", "AnalysisType", etc.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No extra characters are allowed at the end of a line, after the carriage return and line feed. Enclose each data value with double quotes (text qualifier) and separate each field value with a **tab** character (tab delimiter). Data fields with no information (null) may be represented by two consecutive tabs. For example, in the Sample Analysis table, since the "Collected", "ShippingBatchID", and "Temperature" fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields ("ProjectNumber" and "ProjectName") are omitted in this example.

…"LCSW100598" "AQ" "LCSW100598" "LCS" "8270C",…etc.

If a field is populated with less than the maximum allowed number of characters, do not pad the values with leading or trailing spaces. In the above example, although the "MatrixID" field can accommodate up to 10 characters, only 2 characters were entered in this field. **Do not include the delimiter (tab character) within any of the field values.** Example EDD files may be downloaded from the LEDD Checker application.

An example database shall be sent for review prior to the first electronic deliverable in the required .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable should be directed to LabSupport@tetratech.com.

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field. Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01DL and MW01RE are not allowed. Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs.	Text	25	X
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list.	Text	25	X
AnalysisType	Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts.	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and that the LabSampleID must be distinct for a given field sample or lab QC sample and method. Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc.	Text	25	X
LabID	Identification of the laboratory performing the analyses.	Text	7	X
ClientAnalyteID	CAS Number or unique client identifier for an analyte or isotope.	Text	12	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes. For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.) For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID.			
AnalyteName	Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as TIC's).	Text	60	X
Result	Result value for the analyte or isotope. Entries must be numeric. For non-detects of target analytes or isotopes and spikes, **do not enter "ND" or "0". Do not leave this field blank**. If an analyte or spike was not detected, enter the reporting limit value corrected for dilution and percent moisture as applicable. Do not enter "0"	Numeric (1)	20(6)	X
ResultUnits	The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed.	Text	10	X
LabQualifiers	A string of single letter result qualifiers assigned by the lab based on client-defined rules and values. <u>The "U" Lab Qualifier must be entered for all non-detects.</u> Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard values table may be used. If so, these must be added to the standard value table in the application.	Text	7	Q

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
DetectionLimit	For radiochemistry methods, the minimum detectable activity for the isotope being measured. For all other methods: The minimum detection limit value for the analyte being measured.	Numeric (1)	10(6)	X
DetectionLimitType	Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.).	Text	10	X
RetentionTime or Error	For radiochemistry methods only, enter the 2 Sigma Counting Errors. The units for error are entered in the ResultUnits field. For GC/MS methods only, enter the time expressed in decimal minutes between injection and detection for GC/MS TICs only For target analytes in all other methods, leave this field blank. Note: GC retention times are not evaluated at this time.	Text	5	T
AnalyteType	Defines the type of result, such as tracer, surrogate, spike, or target compound.	Text	7	X
PercentRecovery	For radiochemistry methods: The tracer yield, if applicable. For all other analytical methods: The percent recovery value of a spiked compound or surrogate. If the spike or surrogate was not recovered because of dilution, enter "DIL". If a spike or surrogate was not recovered because of matrix interference, enter "INT". If a spike or surrogate was not recovered because it was not added to the sample, enter "NS".	Numeric (1)	5(3)	R
RelativePercentDifference	The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.	Numeric (1)	5(3)	D
ReportingLimit	Reporting limit value for the measured analyte or isotope Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in the project library or QAPP.	Numeric (1)	10(6)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ReportingLimitType	Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each method and matrix is specified in the project library or QAPP.	Text	10	X
ReportableResult	This field indicates whether or not the laboratory chooses an individual analyte or isotope result as reportable. Enter "YES" if the result is reportable. Enter "NO" if the result is not reportable. If only one analysis is submitted for a particular sample and method, enter "YES" for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds (where Analyte Type = TIC). If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter "YES" from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial, undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter "NO" for benzene and enter "YES" for all other compounds. For the diluted analysis (Analysis Type = DL), enter "YES" for benzene and enter "NO" for all other compounds. For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for <u>all</u> TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter "YES" for all TICS. In the other analysis enter "NO" for all TICs. Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given sample, method, and, matrix.	Text	3	X
SpkConcnAdded	The spike added. This value must be reported in the same units as the result.	Numeric (1)	10(6)	R

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	Where (SA) in the following equation: % Recovery = (SSA-SC)/SA x 100% where : SSA is the spiked sample concentration (amount) after spiking. SC is the sample concentration (amount) before spiking. SA is the the expected increase in sample concentration (amount) as a result of spiking. This value must incorporate all correction factors such as dilution factor and moisture content that are applied to the spiked sample when computing the spiked sample concentration or amount.			
SpkParentSampleID	The sampleID of a sample (often called the original sample) that receives a spike aliquot to form a spiked sample such as a matrix spike. This is not the same as the ID of the spiked sample (such as a matrix spike) after spiking. The result for SpkParentSampleID and the result (i.e., SpkConcnAdded) for the spiked sample are used to compute percent recovery of the analyte.	Text	25	R
SamplePrepInitial	The initial sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
SamplePrepFinal	The final sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
Comment	Add any comments or additional information specific to the analyte test result data record.	Text	200	

X Required field.
Q Only required if laboratory has qualified the result.
T Only required for tentatively identified compounds by GC/MS.
R Only required if Analyte_Type =”SPK” or “SURR”.
D Only required for spiked compounds in the LCSD and MSD.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ProjectNumber	Project number assigned by the client.	Text	30	X
ProjectName	Project name assigned by the client.	Text	90	X
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01DL and MW01RE are not allowed Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs.	Text	25	X
Collected	Date and Time of sample collection. Refer to the date/time format at the end of this table. Leave this field blank for Method Blank, LCS, and LCSD	Date/ Time	16*	C
MatrixID	Sample matrix (i.e., AQ, SO, etc.)	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method.	Text	25	X
QCType	This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). For regular environmental samples, populate this field with "NM".	Text	10	X
ShippingBatchID	Unique identifier assigned to a cooler or shipping container used to transport client or field samples. Links all samples to a cooler or shipping container. No value is required for method blanks, LCS, and LCSD.	Text	25	C

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
Temperature	Temperature (in centigrade degrees) of the sample as received. This field is not required for radiochemistry methods.	Numeric (1)	10(6)	C
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a SOP number, however; values used for Laboratory Method IDs are specified by the project and must in the in standard value list for method IDs.	Text	25	X
PreparationType	Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.) For analytical procedures that do not have a specific preparation method number, use “Gen Prep”.	Text	25	X
AnalysisType	Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts.	Text	10	X
Prepared	For radiochemistry leave this field blank. For all other methods enter the date and time of sample preparation or extraction. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Analyzed	Date and time of sample analysis. Refer to the date and time format at the end of this table.	Date/ Time	16*	X
LabID	Identification of the laboratory performing the analysis.	Text	7	X
QCLevel	The level of laboratory QC associated with the analysis reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted for the sample, enter “COA”. If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter “COCAL”	Text	6	X
ResultBasis	Indicates whether results associated with this sample records are reported as wet or percent moisture corrected. This field is only required for soils and sediments. Enter “WET” if results are not corrected for percent moisture. Enter “DRY” if percent moisture	Text	3	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	correction is applied to results.			
TotalOrDissolved	This field indicates if the results related to this sample record are reported as a total or dissolved fraction. This field is only required for metal methods. For all other methods leave this field blank.	Text	3	X
Dilution	Dilution of the sample aliquot. Enter "1" for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution.	Numeric (1)	10(6)	X
HandlingType	Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET). Leave this field blank if the sample analysis was not performed on a leachate.	Text	10	L
HandlingBatch	Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks. Leave this field blank if the sample analysis was not performed on a leachate	Text	12	L
LeachateDate	Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table. Leave this field blank if the sample analysis was not performed on a leachate	Date /Time	16*	L
Percent_Moisture	Percent of sample composed of water. Enter for soil and sediment samples only.	Numeric (1)	10(6)	X
MethodBatch	Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples.	Text	12	X
PreparationBatch	Unique laboratory identifier for a batch of samples prepared together for analysis by one method and	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	treated as a group for method blank, LCS and LCSD association. The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated samples.			
RunBatch	For radiochemistry methods leave this field blank. For all other methods the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2. This field is only required if the A2 table is included with the EDD.	Text	12	X
AnalysisBatch	For radiochemistry methods leave this field blank. For all other methods the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated continuing calibration records in the Laboratory Instrument table. This field is only required if the A2 table is included	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	with the EDD.			
LabReportingBatch	Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records.	Text	12	X
LabReceipt	Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
LabReported	Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Comment	Add any comments or additional information specific to the sample analysis data record.	Text	200	

C Only required for regular samples, duplicates and MS/MSDs.
L Only required if analysis performed is SPLP, TCLP or WET extracts.
X Required field.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.
* Format Date and Time as MM/DD/YYYY hh:mm; where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes.

3.0 Laboratory Data Checker

The Laboratory Data Checker is a web-based application that will review Laboratory Electronic Data Deliverables (LEDDs) for adherence to Tetra Tech's EDD format requirements. EDDs will be reviewed for elements such as missing data and/or columns of data, and compliance of the data within each column to the required data types/lengths. Once an EDD passes through the checker with no errors, it must be submitted to Tetra Tech through the LEDD Checker application.

Access to the LEDD Checker application will be provided by an initial registration/approval process. An Information Systems Group (ISG) Administrator will approve requests for access**.** To access the site or begin the registration process, visit the ISG web site at http://isg.ttnus.com and select the "Laboratory Checker" link on the left of the home page. Registered users may access the checker immediately by logging in to the system using theircredentials. New users must select the "Register" button and provide all of the requested information**.**

After completing all fields on the registration form, select the "Submit" button to complete the request process. Upon verification by an ISG Administrator, an email notification will be sent verifying the user ID, password and account status. Forgotten passwords may be retrieved by using the "Forgot password?" link on the login page. Note that the email address that was provided for registration or password retrieval is the user ID and must be a valid e-mail address.

The general process for submitting EDD files through the LEDD Checker involves a 3-stage process that includes an upload stage, an error checking stage and a submittal stage.

Log into the LEDD Checker by typing your login credentials and select the "Login" button. The LEDD Checker home page provides a general overview of the checker functionality and EDD file format requirements. At the bottom of the home page, example EDDs are provided that may be viewed or downloaded. To download the files, right click on the link and select "Save target as" from the menu. Each LEDD Checker page includes a navigation bar with links to return to the home page or continue the checking and submittal process. Users should **NOT** use the back or forward buttons on the browser, instead use the links provided in the application to navigate through the site.

Detailed information regarding EDD preparation, formatting requirements and text file naming conventions are provided in the Electronic Data Format Requirements Section of the Laboratory SOW.

Begin the upload stage by selecting the "Upload/Check Files" link on the home page. Follow the steps on the upload page starting with the selection of the laboratory name that corresponds to your organization. If your organization is not listed, contact LabSupport@tetratech.com, and provide a full description of your organization name, contact information and include "Laboratory Contractor ID Request" in the subject line. An ISG Administrator will respond to the request via e-mail.

Load the appropriate A1, A2, or A3 target EDD files by clicking the "Browse" button next to each data table input box. A file browser dialog will appear allowing files to be selected from a local or network drive. After the EDD files are loaded, click the "Upload" button to complete the upload stage. Note that each table may be uploaded and checked separately; however, a minimum of the A1 and A3 files are required in order to submit the EDDs.

If the file upload was successful, the checking page will immediately load. Begin the checking stage by selecting the "Check Files" button. The LEDD Checker will begin validating the EDD files for compliance. Depending on file size and network activity the validation process may take several minutes. The progress should be displayed in the information bar at the bottom of the browser window. **Do not** select the "Check Files" button again or otherwise use the browser during this process. Other applications may be used; however, note that the LEDD Checker may not sit idle for more than 30 minutes. If the time is exceeded a new session must be started in a new browser window.

Any errors will be processed and returned on the error page. The following general errors may be returned.

- Column count / table structure errors – due to column header names being included, improper delimiter, extra tabs, extra or missing columns of data, spaces or other characters at the end of a row.
- Row and column value specific errors – may occur for one or more reasons including: data truncation, invalid date / time format, invalid decimal precision or field width exceedance, or if a value is not in a list of valid values or expected range.

If column count / table structure errors are encountered, the LEDD Checker will return an error and stop the checking process.

The EDDs will not be processed any further until the column errors are resolved. Text fields are validated for truncation. Date / Time fields are validated for truncation and format compliance. Numeric decimal fields are validated for truncation, character type compliance and decimal precision. All required fields are validated for null values or empty text strings (i.e. spaces). The LEDD Checker will return a list of all errors in and include a reference to the row number on which the error occurred. Note that consecutive EDD files may be loaded and checked, and submitted while logged in. However, no data may be submitted until all EDD files have passed through the LEDD Checker without errors. The list of errors may be printed by selecting the "Print this Page" button from the checker error page.

If the EDD files pass with no errors, the submittal page will immediately load. To complete the submittal stage, include the following information in the comment and additional information area of the form: laboratory name, laboratory contact person, project name, project number, site name/number, fractions included and any specific comments related to the EDD. Select the “Submit Files” button to continue the submittal process.

The submittal stage is not considered complete until a unique ticket key reference is returned in the browser window. The ticket key reference must be printed for record of submission and future reference. In addition, a copy of the ticket key reference must be included in the PDF data package.

APPENDIX E

ANALYTICAL STANDARD OPERATING PROCEDURES AND CERTIFICATES

LABORATORY ACCREDITATION BUREAU

Certificate Number L2223

Certificate of Accreditation

Accredited to DoD ELAP and ISO/IEC 17025:2005

Katahdin Analytical Services, Inc.

600 Technology Way
Scarborough, ME 04074

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: November 4, 2012

R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 4th of November, 2009

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements

Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Scope of Accreditation For Katahdin Analytical Services

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
1- 207-874-2400

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Katahdin Analytical Services to perform the following tests:

Accreditation granted through: **November 4, 2012**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDE
GC/ECD	608 / 8081A,B / SOM01.2	4 4`-DDT
GC/ECD	608 / 8081A,B / SOM01.2	Aldrin
GC/ECD	608 / 8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	608 / 8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	608 / 8081A,B / SOM01.2	delta-BHC
GC/ECD	608 / 8081A,B / SOM01.2	Dieldrin
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan I
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan II
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	608 / 8081A,B / SOM01.2	Endrin
GC/ECD	608 / 8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor epoxide

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	608 / 8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3’ 4 4’ 5 5’-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3’ 4 4’ 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	8082/8082A	3 4 4’ 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B/C	Diesel range organics (DRO)
GC/FID	8015B/C	Gasoline range organics (GRO)
GC/FID	8011 / 504	1 2-Dibromoethane (EDB)
GC/FID	8011 / 504	1 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene
GC/MS	8260B,C / 524.2	1 1 1 2-Tetrachloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 1-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethene
GC/MS	8260B,C / 524.2	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromoethane (EDB)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloropropane
GC/MS	8260B,C / 524.2	1 3 5-Trimethylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 3-Dichlorobenzene
GC/MS	8260B,C / 524.2	1 3-Dichloropropane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C / 524.2	2 2-Dichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2 / 524.2	2-Butanone
GC/MS	624 / 8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C / 524.2	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	2-Hexanone
GC/MS	8260B,C / 524.2	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	4-Methyl-2-pentanone
GC/MS	8260B,C / SOM01.2 / 524.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	624 / 8260B,C	Acrolein
GC/MS	624 / 8260B,C / 524.2	Acrylonitrile
GC/MS	8260B,C / 524.2	Allyl chloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Benzene
GC/MS	8260B,C / 524.2	Bromobenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Bromochloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromodichloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromoform
GC/MS	8260B,C / SOM01.2 / 524.2	Carbon disulfide
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Carbon tetrachloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2 / 524.2	cis-1 2-Dichloroethene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dibromochloromethane
GC/MS	8260B,C / 524.2	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dichlorodifluoromethane
GC/MS	8260B,C / 524.2	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / 524.2	Ethyl methacrylate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C / 524.2	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2 / 524.2	Isopropyl benzene
GC/MS	8260B,C / SOM01.2 / 524.2	m p-xylenes
GC/MS	8260B,C / 524.2	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl bromide (Bromomethane)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C / 524.2	Methyl methacrylate
GC/MS	8260B,C / SOM01.2 / 524.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methylene chloride
GC/MS	8260B,C / 524.2	Naphthalene
GC/MS	8260B,C / 524.2	n-Butylbenzene
Gc/ms	8260B,C / 524.2	n-Propylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	o-Xylene
GC/MS	8260B,C / 524.2	p-Isopropyltoluene
GC/MS	8260B,C / 524.2	Propionitrile
GC/MS	8260B,C / 524.2	sec-butylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C / 524.2	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	8260B,C / 524.2	Tetrahydrofuran
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Toluene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 2-Dichloroethylene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C / 524.2	trans-1 4-Dichloro-2-butuene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichloroethene (Trichloroethylene)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Vinyl chloride
GC/MS	624 8260B,C	Xylene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	625 / 8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	625 / 8270C,D / SOM01.2	2-Chlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	625 / 8270C,D / SOM01.2	3 3'-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	625 / 8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	625 / 8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	7,12-Dimethylphenethylamine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthene
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthylene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline
GC/MS	625 / 8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	625 / 8270C,D	Benzidine
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	625 / 8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	625 / 8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	625 / 8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	625 / 8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	625 / 8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	625 / 8270C,D / SOM01.2	Fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Fluorene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	625 / 8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D	Isodrin
GC/MS	625 / 8270C,D / SOM01.2	Isophorone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methy methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	625 / 8270C,D / SOM01.2	Naphthalene
GC/MS	625 / 8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	625 / 8270C,D / SOM01.2	Phenanthrene
GC/MS	625 / 8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	625 / 8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
HPLC	8330/8330A/8330B	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B	2-Nitrotoluene
HPLC	8330/8330A/8330B	3-Nitrotoluene
HPLC	8330/8330A/8330B	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B	4-Nitrotoluene
HPLC	8330/8330A/8330B	Hexahydro-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B	Nitrobenzene
HPLC	8330/8330A/8330B	Nitroglycerin
HPLC	8330/8330A/8330B	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B	Tetryl

Non-Potable Water		
Technology	**Method**	**Analyte**
CVAA	245.1 / 7470A / ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	200.7 / 6010B,C / ILM05.3	Aluminum
ICP	200.7 / 6010B,C / ILM05.3	Antimony
ICP	200.7 / 6010B,C / ILM05.3	Arsenic
ICP	200.7 / 6010B,C / ILM05.3	Barium
ICP	200.7 / 6010B,C / ILM05.3	Beryllium
ICP	200.7 / 6010B,C	Boron
ICP	200.7 / 6010B,C / ILM05.3	Cadmium
ICP	200.7 / 6010B,C / ILM05.3	Calcium
ICP	200.7 / 6010B,C / ILM05.3	Chromium
ICP	200.7 / 6010B,C / ILM05.3	Cobalt
ICP	200.7 / 6010B,C / ILM05.3	Copper
ICP	200.7 / 6010B,C / ILM05.3	Iron
ICP	200.7 / 6010B,C / ILM05.3	Lead
ICP	200.7 / 6010B,C / ILM05.3	Magnesium
ICP	200.7 / 6010B,C / ILM05.3	Manganese
ICP	200.7 / 6010B,C	Molybdenum
ICP	200.7 / 6010B,C / ILM05.3	Nickel
ICP	200.7 / 6010B,C / ILM05.3	Potassium
ICP	200.7 / 6010B,C / ILM05.3	Selenium
ICP	200.7	Silicon
ICP	200.7 / 6010B,C / ILM05.3	Silver
ICP	200.7 / 6010B,C / ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	200.7 / 6010B,C / ILM05.3	Thallium
ICP	200.7 / 6010B,C	Tin
ICP	200.7 / 6010B,C	Titanium
ICP	200.7 / 6010B,C / ILM05.3	Vanadium
ICP	200.7 / 6010B,C / ILM05.3	Zinc
ICP/MS	200.8 / 6020/6020A / ILM05.3	Aluminum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Antimony
ICP/MS	200.8 / 6020/6020A / ILM05.3	Arsenic
ICP/MS	200.8 / 6020/6020A / ILM05.3	Barium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Beryllium
ICP/MS	200.8 / 6020/6020A	Boron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cadmium
ICP/MS	200.8 / 6020/6020A	Calcium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Chromium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cobalt
ICP/MS	200.8 / 6020/6020A / ILM05.3	Copper
ICP/MS	200.8 / 6020/6020A	Iron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Lead
ICP/MS	200.8 / 6020/6020A / ILM05.3	Magnesium
ICP/MS	200.8 / 6020/6020A	Manganese

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	200.8 / 6020/6020A	Molybdenum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Nickel
ICP/MS	200.8 / 6020/6020A	Potassium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Selenium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silicon
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silver
ICP/MS	200.8 / 6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Thallium
ICP/MS	200.8 / 6020/6020A	Tin
ICP/MS	200.8 / 6020/6020A	Titanium
ICP/MS	200.8	Uranium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Vanadium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Zinc
IC	300.0 / 9056/9056A	Bromide
IC	300.0 / 9056/9056A	Chloride
IC	300.0 / 9056/9056A	Nitrate as N
IC	300.0 / 9056/9056A	Nitrite as N
IC	300.0 / 9056/9056A	Nitrate + Nitrite
IC	300.0 / 9056/9056A	Orthophosphate as P
IC	300.0 / 9056/9056A	Sulfate
Titration	310.2 / 2320B	Alkalinity
Caculation	2340C	Hardness
Gravimetric	1664A	Oil and Grease
Gravimetric	2540 B, C, D	Solids
ISE	120.1 / 2510 B	Conductivity
ISE	2520B	Practical Salinity
ISE	4500F- C	Fluoride
ISE	4500H+ B	pH
ISE	5210B	TBOD / CBOD
Physical	1010 A	Ignitability
Physical	9040C	pH
Titration	2340B	Hardness
Titration	4500SO_3 B	Sulfite
Titration	9034 / 4500S^{2-} E	Sulfide
Titration	Chap. 7.3.4	Reactive Sulfide
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	180.1 / 2130B	Turbidity
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	335.4 / 9012B / 4500-CN G	Amenable cyanide
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	3500Fe D	Ferrous Iron
UV/VIS	351.2	Kjeldahl nitrogen - total
UV/VIS	353.2 / 4500NO3 F	Nitrate + Nitrite

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/VIS	353.2 / 4500NO3 F	Nitrate as N
UV/VIS	353.2 / 4500NO3 F	Nitrite as N
UV/VIS	365.1 / 4500P E	Orthophosphate as P
UV/VIS	365.4	Phosphorus total
UV/VIS	376.3	AVS-SEM
UV/VIS	410.4	COD
UV/VIS	420.1 / 9065	Total Phenolics
UV/VIS	4500Cl G	Total Residual Chlorine
UV/VIS	5540C	MBAS
UV/VIS	7196A / 3500-Cr D	Chromium VI
UV/VIS	9012B / ILM05.3/ 335.4	Total Cyanide
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
Preparation	**Method**	**Type**
Cleanup Methods	3640A	Gel Permeation Clean-up
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3660B	Sulfur Clean-Up
Cleanup Methods	3665A	Sulfuric Acid Clean-Up
Organic Preparation	3510C	Separatory Funnel Extraction
Organic Preparation	3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	3010A	Hotblock
Volatile Organic Preparation	5030B,C	Purge and Trap

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	8081A,B / SOM01.2	4 4`-DDE
GC/ECD	8081A,B / SOM01.2	4 4`-DDT
GC/ECD	8081A,B / SOM01.2	Aldrin
GC/ECD	8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	8081A,B / SOM01.2	delta-BHC
GC/ECD	8081A,B / SOM01.2	Dieldrin
GC/ECD	8081A,B / SOM01.2	Endosulfan I
GC/ECD	8081A,B / SOM01.2	Endosulfan II
GC/ECD	8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	8081A,B / SOM01.2	Endrin
GC/ECD	8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Heptachlor
GC/ECD	8081A,B / SOM01.2	Heptachlor epoxide
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1016 (PCB-1016)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3’ 4 4’ 5 5’-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3’ 4 4’ 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8082/8082A	3 4 4' 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B,C	Diesel range organics (DRO)
GC/FID	8015B,C	Gasoline range organics (GRO)
GC/FID	8011	EDB
GC/FID	8011	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C	1 1 1 2-Tetrachloroethane
GC/MS	8260B,C / SOM01.2	1 1 1-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	8260B,C / SOM01.2	1 1 2-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethylene
GC/MS	8260B,C	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2	1 2-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 2-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 2-Dichloropropane
GC/MS	8260B,C	1 3 5-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 3-Dichlorobenzene
GC/MS	8260B,C	1 3-Dichloropropane
GC/MS	8260B,C / SOM01.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C	2 2-Dichloropropane
GC/MS	8260B,C / SOM01.2	2-Butanone
GC/MS	8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2	2-Hexanone
GC/MS	8260B,C	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2	4-Methyl-2-pentanone

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	8260B,C	Acrolein
GC/MS	8260B,C	Acrylonitrile
GC/MS	8260B,C	Allyl chloride
GC/MS	8260B,C / SOM01.2	Benzene
GC/MS	8260B,C	Bromobenzene
GC/MS	8260B,C / SOM01.2	Bromochloromethane
GC/MS	8260B,C / SOM01.2	Bromodichloromethane
GC/MS	8260B,C / SOM01.2	Bromoform
GC/MS	8260B,C / SOM01.2	Carbon disulfide
GC/MS	8260B,C / SOM01.2	Carbon tetrachloride
GC/MS	8260B,C / SOM01.2	Chlorobenzene
GC/MS	8260B,C / SOM01.2	Chloroethane
GC/MS	8260B,C / SOM01.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2	cis-1 2-Dichloroethene
GC/MS	8260B,C / SOM01.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	8260B,C / SOM01.2	Dibromochloromethane
GC/MS	8260B,C	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2	Dichlorodifluoromethane
GC/MS	8260B,C	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / SOM01.2	EDB
GC/MS	8260B,C	Ethyl methacrylate
GC/MS	8260B,C / SOM01.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol
GC/MS	8260B,C / SOM01.2	Isopropyl benzene
GC/MS	SOM01.2	m p-xylenes
GC/MS	8260B,C	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	8260B,C / SOM01.2	Methyl bromide (Bromomethane)
GC/MS	8260B,C / SOM01.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C	Methyl methacrylate
GC/MS	8260B,C / SOM01.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	8260B,C / SOM01.2	Methylene chloride
GC/MS	8260B,C	Naphthalene
GC/MS	8260B,C	n-Butylbenzene
GC/MS	8260B,C	n-proplybenzene
GC/MS	8260B,C	o-Xylene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8260B,C	p-Isopropyltoluene
GC/MS	8260B,C	Propionitrile
GC/MS	8260B,C	sec-butylbenzene
GC/MS	8260B,C / SOM01.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	8260B,C / SOM01.2	Tetrachloroethylene (Perchloroethylene)
GC/MS	8260B,C	Tetrahydrofuran
GC/MS	8260B,C / SOM01.2	Toluene
GC/MS	8260B,C / SOM01.2	trans-1 2-Dichloroethylene
GC/MS	8260B,C / SOM01.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C	Trans-1 4-Dichloro-2-butuene
GC/MS	8260B,C / SOM01.2	Trichloroethene (Trichloroethylene)
GC/MS	8260B,C / SOM01.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	8260B,C / SOM01.2	Vinyl chloride
GC/MS	8260B,C	Xylene
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	8270C,D / SOM01.2	2-Chlorophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	8270C,D / SOM01.2	Acenaphthene
GC/MS	8270C,D / SOM01.2	Acenaphthylene
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	8270C,D	Benzidine
GC/MS	8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	8270C,D / SOM01.2	Fluoranthene
GC/MS	8270C,D / SOM01.2	Fluorene
GC/MS	8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	8270C,D	Isodrin
GC/MS	8270C,D / SOM01.2	Isophorone
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methyl methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	8270C,D / SOM01.2	Naphthalene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	8270C,D/ SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	8270C,D / SOM01.2	Phenanthrene
GC/MS	8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
GC/MS	8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine
GC/MS	8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	8270C,D / SOM01.2	2 4-Dinitrophenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D / SOM01.2	3 3`-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D	7,12-Dimethylphenethylamine
HPLC	8330/8330A/8330B (Analysis Only)	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	3-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	Hexahydr-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B (Analysis Only)	Nitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	Nitroglycerin
HPLC	8330/8330A/8330B (Analysis Only)	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B (Analysis Only)	Tetryl
CVAA	7471B/ ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	6010B,C /ILM05.3	Aluminum
ICP	6010B,C /ILM05.3	Antimony
ICP	6010B,C /ILM05.3	Arsenic
ICP	6010B,C /ILM05.3	Barium
ICP	6010B,C /ILM05.3	Beryllium
ICP	6010B,C	Boron
ICP	6010B,C /ILM05.3	Cadmium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP	6010B,C /ILM05.3	Calcium
ICP	6010B,C /ILM05.3	Chromium
ICP	6010B,C /ILM05.3	Cobalt
ICP	6010B,C /ILM05.3	Copper
ICP	6010B,C /ILM05.3	Iron
ICP	6010B,C /ILM05.3	Lead
ICP	6010B,C /ILM05.3	Magnesium
ICP	6010B,C /ILM05.3	Manganese
ICP	6010B,C	Molybdenum
ICP	6010B,C /ILM05.3	Nickel
ICP	6010B,C /ILM05.3	Potassium
ICP	6010B,C /ILM05.3	Selenium
ICP	200.7	Silicon
ICP	6010B,C /ILM05.3	Silver
ICP	6010B,C /ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	6010B,C /ILM05.3	Thallium
ICP	6010B,C	Tin
ICP	6010B,C	Titanium
ICP	6010B,C /ILM05.3	Vanadium
ICP	6010B,C /ILM05.3	Zinc
ICP/MS	6020/6020A / ILM05.3	Aluminum
ICP/MS	6020/6020A / ILM05.3	Antimony
ICP/MS	6020/6020A / ILM05.3	Arsenic
ICP/MS	6020/6020A / ILM05.3	Barium
ICP/MS	6020/6020A / ILM05.3	Beryllium
ICP/MS	6020/6020A	Boron
ICP/MS	6020/6020A / ILM05.3	Cadmium
ICP/MS	6020/6020A	Calcium
ICP/MS	6020/6020A / ILM05.3	Chromium
ICP/MS	6020/6020A / ILM05.3	Cobalt
ICP/MS	6020/6020A / ILM05.3	Copper
ICP/MS	6020/6020A	Iron
ICP/MS	6020/6020A / ILM05.3	Lead
ICP/MS	6020/6020A / ILM05.3	Magnesium
ICP/MS	6020/6020A	Manganese
ICP/MS	6020/6020A	Molybdenum
ICP/MS	6020/6020A / ILM05.3	Nickel
ICP/MS	6020/6020A	Potassium
ICP/MS	6020/6020A / ILM05.3	Selenium
ICP/MS	6020/6020A / ILM05.3	Silver
ICP/MS	6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	6020/6020A / ILM05.3	Thallium
ICP/MS	6020/6020A	Tin

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/MS	6020/6020A	Titanium
ICP/MS	6020/6020A / ILM05.3	Vanadium
ICP/MS	6020/6020A / ILM05.3	Zinc
IC	9056/9056A	Chloride
IC	9056/9056A	Fluoride
IC	9056/9056A	Nitrate as N
IC	9056/9056A	Nitrite as N
IC	9056/9056A	Sulfate
Gravimetric	9070A / 9071B	Oil and Grease
Physical	1010A	Ignitability
Physical	9045D	pH
Titration	Chap 7.3.4	Reactive Sulfide
TOC	Lloyd Kahn	Total organic carbon
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
UV/VIS	376.3	AVS-SEM
UV/VIS	3500Fe D	Ferrous Iron
Cleanup Methods	3630C	Silica Gel
UV/VIS	7196	Chromium VI
UV/VIS	7196A	Chromium VI
UV/VIS	9012B / ILM05.3	Total cyanide
Preparation	**Method**	**Type**
Preparation	1311	Toxicity Characteristic Leaching Procedure
Preparation	1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	3660B	Sulfur
Cleanup Methods	3620C	Florsil
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3640A	GPC
Organic Preparation	3540C	Soxhlet Extraction
Organic Preparation	3545A	Pressurized Fluid Extraction
Organic Preparation	3550C	Sonication
Inorganics Preparation	3050B	Hotblock
Inorganics Preparation	3060A	Alkaline Digestion
Volatile Organics Preparation	5035/5035A	Closed System Purge and Trap

Notes:

1) This laboratory offers commercial testing service.

Approved By: _____ Date: November 4, 2009

R. Douglas Leonard
Chief Technical Officer

Issued: 11/04/09

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Inductively Coupled Plasma Atomic Emission Spectroscopy
Perkin Elmer Optima-3000/3300DV/5300DV

SW-846 Method 6010C

APPROVALS:

Area Supervisor: Denise Coffey Date: 1-19-09
Denise S. Coffey

QA Officer: Tom C. Boocher Date: 1-16-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 1/19/09
Jeff P. Glaser

Procedure Number: GR-01-100
Revision Number: 5.7

Date Initiated: 4/4/96
Effective Date: 2/5/09

Date Revised: 1/16/09
Pages Revised: All

By: Denise S. Coffey

Total Number of Pages: 47

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma atomic emission spectroscopy (ICP) determines a wide range of elements in solution.

1.2 The procedure is applicable to a variety of matrices, including but not limited to soil, water, TCLP leachates, EPTOX leachates, SPLP leachates, ASTM leachates, oil, solvents, paint, sludge, pure product or any other matrix that may be extracted or dissolved into an acidic aqueous solution.

1.3 Most matrices require solubilization and/or digestion prior to ICP analysis.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Method 6010C, "Inductively Coupled Plasma-Atomic Emission Spectrometry", Revision 3, February 2007

2.2 *ICP Winlab Software Guide*, Revision E, June 1997

2.3 *Optima 3000 Family Hardware Guide*, Release B, August 1997

3.0 SUMMARY OF PROCEDURE

3.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods. Refer to the appropriate SW-846 3000 series methods for this procedure.

3.2 This procedure describes the simultaneous, multi-elemental determination of elements by ICP. The procedure measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the argon plasma torch via an argon carrier gas. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by an eschelle grating polychrometer, and the intensities are monitored by a Segmented Charge Coupled Device (SCD).

3.3 Tables 1 and 1A specify applicable elements, primary wavelengths, and reporting limits. The wavelengths listed in these tables have been selected because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity, and are treated with the same corrective techniques for spectral interference. Other elements may be added as more information becomes available, and as required.

3.4 Quantitation limits are sample dependent and may vary as the sample matrix varies; the quantitation limits provided in Tables 1 and 1A are the current nominal limits for relatively interference-free aqueous and soil matrices. These limits may need to be elevated for more complex sample matrices.

3.5 Scandium (Sc) and Yttrium (Y) are used as internal standards and are not of interest at the quantitation limit. Background correction is required for trace element determination. Background intensities must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity must be free from spectral interferences, and reflect the same change in background intensity that

occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

3.5.1 Refer to Attachment 23.1, Table 1, Analytical Wavelenths and Instrumental Reporting Limits for the Optima 3000.

3.5.2 Refer to Attachment 23.2, Table 1A, Analytical Wavelengths and Viewing for Optima 3300DV.

4.0 PARAMETER OR COMPOUND LIST

4.1 This procedure may be used for the analysis of the following metals:

Aluminum	Molybdenum
Antimony	Nickel
Arsenic	Phosphorus
Barium	Potassium
Beryllium	Selenium
Boron	Silicon
Cadmium	Silver
Calcium	Sodium
Cerium**	Strontium
Chromium	Sulfur *
Cobalt	Thallium
Copper	Tin
Iron	Titanium
Lead	Vanadium
Lithium	Zinc
Magnesium	
Manganese	

* Optima 3300DV and 5300DV only
**For interference correction only.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal,* latest revision

5.2 TriMatrix SOP GR-01-137, *Acid Digestion of Sediments, Sludges and Soils Using Block Digestion,* latest revision

5.3 TriMatrix SOP GR-01-147, *Acid Digestion of Wastewater, Dissolved Water, Total Water and Extracts, using Block Digestion for the ICP Analysis of Total Metals,* latest revision

5.4 TriMatrix SOP GR-01-119, *Toxicity Characteristic Leaching Procedure (TCLP),* latest revision

5.5 TriMatrix SOP GR-01-117, *Synthetic Precipitation Leaching Procedure (SPLP),* latest revision

5.6 TriMatrix SOP GR-10-123, *Element™ Data Transfer and Review*, latest revision

5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Spectral Interferences

6.1.1 Spectral interferences are caused by: (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) stray light from the line emission of high-concentration elements.

6.1.2 Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

6.1.3 Users of simultaneous multi-element instruments, such as the Optima 3000, 3300DV and 5300DV must verify the absence of spectral interference at the analytical wavelength. As an example, Table 2 (Refer to Attachment 23.3, Table 2, Analyte Emission Equivalents Arising from Interference for the Optima 3000, Instrument 101) shows the interferences that have been documented for the Optima 3000. The data in Table 2 is intended as rudimentary guides for indicating potential interferences. This data will periodically change; therefore, these values are only guides. Interelement correction factors are re-evaluated every six months, or following major instrument maintenance, whichever is more frequent.

6.1.3.1 The interference is expressed as apparent false concentration per ppm of interferent. The instrument software automatically calculates and compensates for interelement corrections (IEC). For more information on the topic of interelement corrections, refer to the Optima 3000, Optima 3300DV or Optima 5300DV software manuals.

6.1.3.2 The dashes in Table 2 indicate that no measurable interferences were observed at the concentrations used to create this table.

6.1.4 Interelement interference check solution elements are selected based on the target analyte or analytes interfered with.

6.1.4.1 For example, iron interferes with beryllium and is included in solution IFB-1. Titanium interferes with thallium and is included in solution IFB-2.

6.1.4.2 Multiple elements that interfere with a target analyte are in the solution where the highest degree of interference can be evaluated. For example, copper, iron, manganese, nickel and vanadium interfere with antimony. Iron is in IFB-1 and the rest are in IFB-2 so antimony is in IFB-2.

6.1.4.3 To further validate interelement correction factors, all elements are monitored in each IFA solution if a false positive or negative greater then twice the analyte

reporting limit is observed. If any are observed, locate and correct the problem before reporting analyte results.

6.2 Physical Interferences

6.2.1 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations due to a decrease in nebulization and transport efficiencies. Both the Optima 3000 and 3300DV compensate for the viscosity and solids content by using on-line internal standard addition. The internal standards, in this case Sc and Y, are added via a peristaltic pump to the sample stream as it is pumped into the plasma. If the internal standard intensity changes in a sample relative to a standard, the data for that sample is electronically altered to reflect the assumed change in analyte intensity.

6.2.2 Samples containing large amounts of elements may decrease the forward power available to the plasma for elemental excitation. The RF generator used by Perkin Elmer in both the Optima 3000 and 3300DV is a free running generator, meaning that the generator may compensate for a decrease in the forward power by increasing the amount of power to the RF coil. The result of this compensation is that the forward power available to the plasma will remain at the set level if large quantities of analytes are introduced into the system.

6.2.3 The Optima 3000, 3300DV and 5300DV use a mass flow control for the argon supply which has been reported to improve instrument performance.

6.3 Chemical Interferences

6.3.1 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique.

6.3.2 If observed, chemical interferences can be minimized as follows:

6.3.2.1 By careful selection of operating conditions such as incident power and/or observation position

6.3.2.2 By buffering the sample

6.3.2.3 By matrix matching

6.3.2.4 By using the method of standard additions

6.3.3 Chemical interferences are highly dependent on matrix type and the specific element.

7.0 SAFETY PRECAUTIONS

7.1 The analyst must comply with all standard operating procedures for health and safety as outlined in the TriMatrix Laboratories, Inc. Laboratory Safety Manual.

7.2 Concentrated acids are used in the preparation of standards and samples for analysis by ICP. Gloves, labcoats, and safety glasses must be worn at all times when handling concentrated acids. Gloves must also be worn when handling samples. Refer to the Material Safety Data Sheets (MSDS) for information on these or any other chemicals utilized in this procedure.

7.3 Check the exhaust hood over the instrument to be sure it is operating correctly. If the ventilation system is not working properly, extinguish the plasma and immediately contact the metals lab supervisor. Under no circumstances should the ICP be used if the exhaust hood is not working.

7.4 The plasma emits UV radiation. Avoid looking directly at the plasma without some type of strong UV protection. The instrument uses a UV resistant material in the viewing port so the analyst may watch the plasma. Do not tamper with this plate. Do not operate the instrument without this plate in place. Do not attempt to view the plasma directly or indirectly in any way. Failure to follow this policy may cause very serious and immediate damage to the retina of the eye.

7.5 The ICP emits a strong RF field. To minimize exposure to this field, Perkin Elmer has included several safety interlocks to prevent exposure of the analyst to harmful radiation. Never override any interlock on the ICP. When working on the instrument, always replace all of the RF shielding using all of the supplied screws. If any safety device has been tampered with, contact the metals lab supervisor.

7.6 The ICP uses argon to sustain the plasma and to nebulize the sample into the plasma. Although argon in and of itself is not hazardous or flammable, it may cause suffocation through oxygen deprivation. It is therefore imperative that all sources of argon be turned off at the valve when not in use. Since argon is colorless and odorless, if you feel lightheaded, evacuate the metals lab at once and notify the metals lab supervisor.

7.7 Many of the chemicals and elements used in this procedure are toxic if ingested. Refer to the MSDS for information on these or any other chemicals utilized in this procedure.

7.8 No food or drink is allowed in the metals lab. Food or drink may become contaminated with acid or metals and may therefore be hazardous.

7.9 Wash hands before starting work. Chemicals may be present on the skin which may interfere with metals analysis. Wash hands before leaving the metals lab. Chemicals and acids may be on the skin which could eventually be ingested or passed on to a third party through casual contact.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Aqueous samples must have been acidified at the time of collection to a pH of <2. If the pH is not <2 when received by the laboratory, the sample pH must be lowered to <2 during the sample receipt process. A minimum of 24 hours must elapse after adjustment prior to sample digestion.

8.2 Solid samples require no preservation for metals. However, if the same sample container is scheduled for other analyses where refrigeration of the sample is needed, store the sample at 4 ±2° C.

8.3 Samples may be collected in glass or plastic. If Silicon or Boron is to be analyzed, plastic containers must be used. The acidified sample must never come into contact with any metal as this would cause leaching of the metal into the sample.

8.4 For the analysis of dissolved metals, the sample must have been filtered on site and then acidified to pH <2. If the pH was not <2, the sample pH must be lowered to <2 during the sample receipt process and a minimum of 24 hours must elapse after adjustment prior to sample analysis.

8.5 Hold times for all metals for ICP analysis is 180 days. In the case of TCLP or similar leachates, the hold time starts once leachate generation has been completed.

8.6 The minimum sample size for this procedure is 50 mL and 150 mL if samples require Matrix QC. Soils, sediments, and solid waste samples require a minimum of 50 g of sample. This implies that the sample must be solubilized prior to analysis. A smaller minimum amount may be used if the sample may be diluted at the sacrifice of the quantitation limit.

8.7 Digested (solubilized) samples and undigested acidified aqueous samples need not be refrigerated. All samples and digestates must be at room temperature before analysis.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Inductively coupled argon plasma emission spectrometer:

9.1.1 Optima 3000/3300DV/5300DV Inductively Coupled Plasma Optical Emission Spectrometer with an Eschelle based polychrometer and a Segmented-Array Charge-Coupled-Device Detector (SCD). The instrument is capable of taking readings in the UV/VIS spectrum with a wavelength range of 167 to 782 nm. The RF generator employed runs at 40 MHz.

Operating conditions: The analyst must follow the instructions provided by the instrument's manufacturer. For operation with organic solvents, the use of the auxiliary argon inlet is recommended, as is solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental reporting limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. All measurements must be within instrument linear range. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

9.1.2 Perkin Elmer IEEE autosampler, model AS-90/AS-91. This autosampler must be installed by a Perkin Elmer representative.

9.2 Recirculator/chiller, Polyscience 633 or equivalent. Temperature must be maintained at 18° C and pressure must be at 50 psi. The recirculator is attached directly to the instrument to maintain isothermal conditions within the optics cavity.

9.3 Computer running Perkin Elmer control software. An IBM compatible 80486SX25 or faster computer with at least 64 MB RAM, a 1G hard disk, and a VGA color monitor is needed. The current software used by the instrument is ICP Winlab software version 1.47 running in a Windows NT Environment. The software version may be updated if it performs at least as well as the older version. Major updates, such as moving to another operating platform will require an update to this procedure.

9.4 Appropriate Printer. The printer must be attached to the computer using an appropriate communication cable. Refer to the Instrument manual for further printer specifications.

9.5 15 mL clear centrifuge tubes or equivalent. The tubes must stand upright within the autosampler rack without tipping or leaning.

9.6 Autopipettors capable of delivering 50-1000 µL.

9.7 Class A volumetric flasks, various volumes.

9.8 Class A reusable pipettes, various volumes.

9.9 500 mL plastic bottles for standards and quality control.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 At the beginning of each shift (every 8 hours) the manifold pump tubing must be replaced. Refer to Section 15.5 for a discussion on changing the pump tubing. Update the instrument maintenance logbook.

10.2 There are 3 filters on the Optima 3000 that must be cleaned every week. The first two filters are located on the back of the instrument on the right-hand side. The third filter is located on the left.

10.2.1 Remove the filters from the holders. The right-hand filters have a cover that 'pops' off. The left-hand filter just slides up and out of its holder.

10.2.2 Wash the filters in soap and water to loosen any dirt and dust from the filters.

10.2.3 Squeeze any excess water from the filters. Dry the filters with a paper towel. Allow these filters to air dry.

10.2.4 If there are extra filters, replace the filters on the instrument, otherwise wait until the newly washed filters are dry before replacing them.

10.2.5 Place a note in the instrument maintenance logbook that the filters were cleaned.

10.3 Inspect the waste container on the floor under the instrument every shift. If the container is full, properly dispose of the liquid.

10.4 Visually inspect the torch before lighting the plasma. Do not touch or move the torch. This inspection must be visual only.

10.4.1 Open the doors to the torch chamber.

10.4.2 Observe the torch. If the torch is broken or cracked, replace it. Refer to Section 10.6 for a discussion on how to assemble and disassemble the torch.

10.4.3 Listen for any obvious leaks in the argon lines going to the torch or nebulizer. If leaks are heard, investigate them and tighten the fittings to stop the leak. If the leak cannot be found or cannot be stopped, contact the metals lab supervisor.

10.5 Once every four to eight weeks, disassemble the entire torch assembly and inspect all parts for wear. Replace all worn or damaged parts. Record all changes in the instrument maintenance logbook.

10.5.1 Inspect the drain line at the base of the spray chamber. There must be no leaks or cracks in the tubing. If a problem exists, replace the O-rings in the drain cap. Place a note in the instrument maintenance logbook that the tubing was replaced.

10.5.2 Inspect the nebulizer for wear.

10.5.14.1 Salt deposits, if present, should be removed with water.

10.5.14.2 If a cross-flow nebulizer is used, check the tips for wear. Replace the tips if excessive wear is noted. Record any actions in the instrument maintenance logbook.

10.5.3 Replace the alumina injector with a newly cleaned one. To clean dirty alumina injectors, place them in a plastic 100 mL graduated cylinder. In a hood, cover the injectors with aqua regia. Allow the injectors to soak for 2 hours. Rinse the alumina injectors with tap water, then ASTM Type II water. Place clean injectors in the appropriate drawer for future use. Record the change in the instrument maintenance logbook.

10.5.4 Check all O-rings on the alumina injector holder. Any cracked or chipped O-rings must be replaced with new ones. The book located next to the spare parts drawers has an exploded view of the torch assembly. Use this diagram to identify the exact part number needed for replacement. Note any replacements in the instrument maintenance logbook.

10.5.5 Check the torch for damage. Brown discoloration at the top of the torch is normal. Since the Optima torch has exit windows cut into the torch for light to pass through, this discoloration need not be rectified. Look at the bottom of the torch. There are frequently chips at this spot on the torch. If chips are observed, replace the torch. Never use a torch that is chipped, cracked, or broken. See the metals lab supervisor for help in replacing the torch as this procedure is beyond the scope of this SOP. Note replacement in the instrument maintenance logbook.

10.5.6 Replace the alumina injector support in the spray chamber.

10.5.7 Re-install the spray chamber and alumina injector holder unit in the quick-change mount.

10.5.8 Replace the alumina injector.

10.5.9 Replace the torch on the assembly.

10.5.10 Replace the nebulizer.

10.5.11 Replace the torch assembly on the mounting bracket.

10.5.12 Close the torch box doors.

10.5.13 Note any changes made to the torch during this procedure in the instrument maintenance logbook.

10.6 Inspect the printing quality of the printer. If the print is hard to read or light in appearance, replace the printer cartridge with the appropriate replacement.

11.0 CHEMICALS AND REAGENTS

11.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Re-distilled acids may be used if it has been demonstrated that the acid is free from contamination.

11.1.1 Concentrated hydrochloric acid, trace metal grade (Fisher Cat #A50B-212) (or equivalent).

11.1.2 Concentrated nitric acid, trace metal grade (Fisher Cat. #A509-212) (or equivalent).

11.2 ASTM Type II water (ASTM D1193). Deionized water is fed into an all glass distillation unit from Barnstead (Model FI-STREEM 2). The resulting distillate is immediately placed into a plastic container. Impurities are measured by the Inorganics group at TriMatrix. This is the only water acceptable for use in the metals laboratory for dilutions or standard preparation.

11.3 Argon gas supply: Welding grade or better. Currently, this is plumbed from a liquid argon tank located outside of the building. Argon is used as the main plasma gas, the auxiliary plasma gas, and the nebulizer carrier gas.

11.4 Nitrogen gas supply: Currently, this is plumbed from a liquid N2 tank located outside of the building. N_2 is used to purge the optics cavity of Oxygen so that measurements below 190 nm may be made accurately.

11.5 Aqua Regia. Three parts hydrochloric to one part nitric. This is used to clean torches and alumina injectors.

12.0 STANDARDS PREPARATION

12.1 All standards are matrix matched as close to sample matrices as possible. Primary Standard stock solutions are purchased from Inorganic Ventures, Absolute Standards and/or SCP Science. All stock solutions are ICP grade single-element (1000 or 10,000 mg/L) or custom multi-element solutions at varied concentrations. All primary standards expire on the supplier's expiration date.

12.2 Secondary Standard stock solutions are purchased from other certified sources. All secondary standards expire on the supplier's expiration date.

12.3 All ICP working standards and dilutions prepared from primary standards expire 3 months after preparation or on the expiration date of the primary standard, whichever is earlier.

12.4 Current purchased custom multi-element standards include: ICP Stock A, ICP Stock B, CRL 1 Stock and

CRL 2 Stock.

12.5 Prepare the working standards.

Note: If Sulfur is a target analyte, refer to Section 12.9.

12.5.1 Rinse four clean 500 mL class A volumetric flasks with ASTM Type II water a minimum of three times.

12.5.2 Place 100 mL ASTM Type II water into each flask.

12.5.3 Pipette the correct amounts of acid into the flasks depending on the final acid concentration desired. Section 12.8.3.1 through Section 12.8.3.3 shows the acid concentrations used for matrix matching.

12.5.3.1 For 10% HCl/5% HNO_3, place 50 mL HCl into the flask and swirl. When the mixing lines are gone from solution, add 25 mL HNO_3 to the flask and swirl until the mixing lines are gone. This is for solid samples.

12.5.3.2 For 5% HCl/5% HNO_3, place 25 mL HCl into each flask and swirl. When the mixing lines are gone from solution, add 25 mL HNO_3 to each flask and swirl until the mixing lines are gone. This is for all digested aqueous samples.

12.5.3.3 For 0.5% HNO_3, place 2.5 mL HNO_3 into each flask and swirl until the mixing lines are gone. This is for non-digested aqueous samples.

12.5.4 Pipette 5.0 mL of ICP Stock A, 5.0 mL of ICP Stock B, 500 µL of 1000 mg/L Co and 500 µL of 1000 mg/L Ag into one of the flasks. This is standard four for the calibration curve.

12.5.5 Pipette 2.5 mL of ICP Stock A, 2.5 mL of ICP Stock B, 250 µL of 1000 mg/L Co and 250 µL of 1000 mg/L Ag into one of the flasks. This is standard three for the calibration curve.

12.5.6 Pipette 0.5 mL of ICP Stock A, 0.5 mL of ICP Stock B, 50 µL of 1000 mg/L Co and 50 µL of 1000 mg/L Ag into one of the flasks. This is standard two for the calibration curve.

12.5.7 Pipette 0.25 mL of CRL 1 Stock and 0.25 mL of CRL 2 Stock into one of the flasks. This is standard one for the calibration curve.

12.5.8 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.5.9 Update the standard numbers in element.

12.5.10 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.6 Sulfur working standards are prepared separately.

12.6.1 Rinse four clean 100 mL class A volumetric flasks with ASTM Type II water a minimum of three times.

12.6.2 Place 25 mL ASTM Type II water into each flask.

12.6.3 Pipette the correct amounts of acid into the flasks depending on the final acid concentration desired.

12.6.3.1 For 10% HCl/5% HNO_3, place 10 mL HCl into the flask and swirl. When the mixing lines are gone from solution, add 5 mL HNO_3 to the flask and swirl until the mixing lines are gone. This is for solid samples.

12.6.3.2 For 5% HCl/5% HNO_3, place 5 mL HCl into each flask and swirl. When the mixing lines are gone from solution, add 5 mL HNO_3 to each flask and swirl until the mixing lines are gone. This is for digested aqueous samples.

12.6.3.3 For 0.5% HNO_3, place 0.5 mL HNO_3 into each flask and swirl until the mixing lines are gone. This is for non-digested aqueous samples.

12.6.4 Pipette 500 µL of the 1000 mg/L sulfur standard into one of the flasks. This is standard 4 for the curve.

12.6.5 Pipette 250 µL of the 1000 mg/L sulfur standard into a second flask. This is standard 3 for the curve.

12.6.6 Pipette 50 µL of the 1000 mg/L sulfur standard into the third flask. This is standard 2 for the curve.

12.6.7 Pipette 10 µL of the 1000 mg/L sulfur standard into the fourth flask. This is standard 1 for the curve.

12.6.8 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.6.9 Update the standard numbers in element.

12.6.10 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.6.11 Refer to Attachment 23.6, Table 5, Optima Working Standard Concentrations.

12.7 Prepare the stock interelement check standard solutions for IFA-1, IFA-2, IFB-1, and IFB-2.

12.7.1 The solution Interf-1 is purchased from Inorganic Ventures.

12.7.2 Prepare solution ICS-1 Analytes.

12.7.2.1 Rinse a clean 100 mL class A volumetric flask with ASTM Type II water a minimum of three times.

12.7.2.2 Place 2 mL HNO_3 in the flask.

12.7.2.3 Pipette the following amounts of the standards below into the flask, swirling after each addition. Always use a new pipette for each standard and never pipette directly out of the primary standard source bottle.

12.7.2.3.1 10 mL each of 1000 mg/L Ag, Cd, Ni, Pb, Zn

12.7.2.3.2 5 mL each of 1000 mg/L Ba, Be, Co, Cr, Cu, Mn, V

12.7.2.4 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.7.2.5 Update the standard numbers in element.

12.7.2.6 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.7.3 Prepare solution IFA-2 Interferent.

12.7.3.1 Rinse a clean 100 mL class A volumetric flask with ASTM Type II water a minimum of three times. Place 10 mL HNO_3 in the flask.

12.7.3.2 Pipette the following amounts of the standards below into the flask, swirling after each addition. Always use a new pipette for each standard, and never pipet directly out of the primary standard source bottle.

10 mL each of 10,000 mg/L Cr, Cu, Mn, Ni, Ti, V

12.7.3.3 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.7.3.4 Update the standard numbers in element.

12.7.3.5 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents..

12.7.4 Prepare solution IFA-2 Analyte.

12.7.4.1 Rinse a clean 100 mL class A volumetric flask with ASTM Type II water a minimum of three times. Place 2 mL HNO_3 in the flask.

12.7.4.2 Pipette the following amounts of the standards below into the flask, swirling after each addition. Always use a new pipette for each standard and never pipet directly out of the primary standard source bottle.

12.7.4.2.1 10 mL each of 1,000 mg/L Al, As, B, Mo, Sb, Se, Tl

12.7.4.2.2 5 mL each of 10,000 mg/L Ca, Mg, Na

12.7.4.3 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.7.4.4 Update the standard numbers in element.

12.7.4.5 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.8 Prepare the interelement check standard solution IFA-1.

12.8.1 Rinse a clean 500 mL Class A volumetric flask with ASTM Type II water a minimum of three times.

12.8.2 Place about 100 mL ASTM Type II water into the flask.

12.8.3 Pipette the correct amounts of acid into the flask depending on the final acid concentration desired.

12.8.3.1 For 10% HCl/5% HNO_3. Place 50 mL HCl into the flask and swirl. When the mixing lines are gone from the solution, add 25 mL HNO_3 to the flask, and swirl until the mixing lines are gone. This is for solid samples.

12.8.3.2 For 5% HCl/5% HNO_3. Place 25 mL HCl into the flask and swirl until the mixing lines are gone from the solution. Add 25 mL HNO_3 to the flask and swirl until the mixing lines are gone from the solution. This is for digested aqueous samples.

12.8.3.3 For 0.5% HNO_3. Place 2.5 mL HNO_3 into the flask and swirl. This is for non-digested aqueous samples.

12.8.4 Pipette 10 mL of the solution Interf-1 into the flask and swirl.

12.8.5 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.8.6 Update the standard numbers in element.

12.8.7 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.9 Prepare the interelement check standard solution IFA-2.

12.9.1 Rinse a clean 500 mL Class A volumetric flask with ASTM Type II water a minimum of three times.

12.9.2 Place 100 mL ASTM Type II water into the flask.

12.9.3 Pipette the correct amount of acid into the flask depending on the final acid concentration desired.

12.9.3.1 For 10% HCl/5% HNO_3. Place 50 mL HCl into the flask and swirl until the mixing lines are gone from the solution. Add 25 mL HNO_3 to the solution. Add 25 mL HNO_3 to the flask and swirl until the mixing lines are gone. This is for solid samples.

12.9.3.2 For 5% HCl/5% HNO_3. Place 25 mL HCl into the flask and swirl until the mixing lines are gone from the solution. Add 25 mL HNO_3 to the flask and swirl until the mixing lines are gone from the solution. This is for digested aqueous samples.

12.9.3.3 For 0.5% HNO_3. Place 2.5 mL HNO_3 into the flask and swirl. This is for non-digested aqueous samples.

12.9.4 Pipette 10 mL of the solution IFA2 – Interferent into the flask.

12.9.5 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.9.6 Update the standard numbers in element.

12.9.7 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.10 Prepare the interelement check standard solution IFB-1.

12.10.1 Rinse a clean 500 mL Class A volumetric flask with ASTM Type II water a minimum of three times.

12.10.2 Place 100 mL ASTM Type II water into the flask.

12.10.3 Pipette the correct amount of acid into the flask depending on the final acid concentration desired.

12.10.3.1 For 10% HCl/15% HNO_3. Place 50 mL HCl into the flask and swirl until the mixing lines are gone from the solution. Add 25 mL HNO_3 to the solution. Add 25 mL HNO_3 to the flask and swirl until the mixing lines are gone. This is for solid samples.

12.10.3.2 For 5% HNO_3/5% HCl. Place 25 mL HCl into the flask and swirl until the mixing lines are gone. This is for solid samples.

12.10.3.3 For 0.5% HNO_3. Place 2.5 mL HNO_3 into the flask and swirl. This is for non-digested aqueous samples.

12.10.4 Pipette 10 mL of the solution Interf-1 into the flask and swirl.

12.10.5 Pipette 5 mL of the solution ICS-1 analyte into the flask and swirl.

12.10.6 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.10.7 Update the standard numbers in element.

12.10.8 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.10.9 Refer to Attachment 23.7, Table 6 IFB-1, Working Solution.

12.11 Prepare the Interelement Check solution IFB-2

12.11.1 Rinse a clean 500 mL class A volumetric flask with ASTM Type II water a minimum of three times.

12.11.2 Place 100 mL ASTM Type II water into a flask.

12.11.3 Pipette the correct amount of acid into the flask depending on the final acid concentration desired.

12.11.3.1 For 10% HCl/5% HNO_3, place 50 mL HCl into the flask and swirl. When the mixing lines are gone from the solution, add 25 mL HNO_3 to the flask, and swirl until the mixing lines are gone. This is for solid samples.

12.11.3.2 For 5% HCl/5% HNO_3, place 25 mL HCl into each flask and swirl. When the mixing lines are gone from solution, add 25 mL HNO_3 to each flask and swirl until the mixing lines are gone. This is for digested aqueous samples.

12.11.3.3 For 0.5% HNO_3, place 25 mL HNO_3 into each flask and swirl until the mixing lines are gone. This is for non-digested aqueous samples.

12.11.4 Pipette 10 mL of the solution IFA-2 interferent into the flask. Swirl the flask.

12.11.5 Pipette 5 mL of the solution IFA-2 analyte into the flask. Swirl the flask.

12.11.6 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.11.7 Update the standard numbers in element.

12.11.8 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.11.9 Refer to Attachment 23.8, Table 7, IFB-2 Working Solution.

12.12 Prepare the CRL working standard.

12.12.1 Rinse a clean 500 mL Class A volumetric flask with ASTM Type II water a minimum of three times.

12.12.2 Place about 100 mL ASTM Type II water into the flask.

12.12.3 Pipette the correct amounts of acid into the flask depending on the final acid concentration desired.

12.12.3.1 For 10% HCl / 5% HNO3. Place 50 mL HCl into the flask and swirl. When the mixing lines are gone form the solution, add 25 mL HNO3 to the flask, and swirl until the mixing lines are gone. This is for solid samples.

12.12.3.2 For 5% HCl / 5% HNO3. Place 25 mL HCl into the flask and swirl. When the mixing lines are gone from the solution, add 25 mL HNO3 to the flask and swirl until the mixing lines are gone. This is for digested aqueous samples.

12.12.3.3 For 0.5% HNO3. Place 2.5 mL HNO3 into the flask and swirl. This is for non-digested aqueous samples.

12.12.4 Pipette 0.5 mL of the CRL 1 Stock and 0.5 mL of the CRL 2 Stock into the flask.

12.11.6 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.11.7 Update the standard numbers in element.

12.11.8 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.13 Prepare the internal standard solution.

12.13.1 Rinse a clean 1000 mL Class A volumetric flask with ASTM Type II water a minimum of three times.

12.13.2 Place 100 mL ASTM Type II water into the flask.

12. 13.3 Pipette the correct amounts of acid into the flask depending on the final acid concentration desired.

12.13.3.1 For 10% HCl / 5% HNO3. Place 100 mL HCl into the flask and swirl. When the mixing lines are gone from the solution, add 50 mL HNO3 to the flask, and swirl until the mixing lines are gone. This is for solid samples.

12.13.3.2 For 5% HCl / 5% HNO3. Place 50 mL HCl into the flask and swirl. When the mixing lines are gone from the solution, add 50 mL HNO3 to the flask and swirl until the mixing lines are gone. This is for digested aqueous samples.

12.13.3.3 For 0.5% HNO3. Place 5.0 mL HNO3 into the flask and swirl. This is for non-digested aqueous samples.

12.13.4 Use the volumes listed to prepare concentrations for the instrument being used:

12.13.4.1 Pipette 0.50 mL of 1000 mg/L Sc stock standard and 1.0 mL of 1000 mg/L Y stock standard into the flask for the Optima 3000.

12.13.4.2 Pipette 2.0 mL of 1000 mg/L Sc stock standard and 0.075 mL of 1000 mg/L Y stock standard into the flask for the Optima 3300DV.

12.13.4.3 Pipette 2.0 mL of 1000 mg/L Sc stock standard and 0.34 mL of 1000 mg/L Y stock standard into the flask for the Optima 5300DV.

12.13.5 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.13.6 Update the standard numbers in element.

12.13.7 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.14 Prepare the Secondary Calibration Verification (SCV)

12.14.1 There are two SCV stock solutions currently being purchased from Environmental Express.

12.14.1.1 QC Solution A:

12.14.1.1.1 500 mg/L of Al, As, P, Tl, Se

12.14.1.1.2 1000 mg/L each of Ca, K, Mg, Na

12.14.1.1.3 100 mg/L each of V, Ba, B, Cu, Fe, Li, Zn, Cd, Cr, Co, Pb, Mn, Ni, Sr

12.14.1.1.4 10 mg/L of Be

12.14.1.2 QC Solution B:

12.14.1.2.1 500 mg/L of Sb, Sn, Si

12.14.1.2.2 100 mg/L each of Ag, Mo, Ti

12.17.2 The working SCV is prepared fresh daily.

12.17.2.1 Pipet 50 µL of each SCV stock into 9.9 mL of 10% HCl/5% HNO_3 reagent blank. This is for solid samples.

12.17.2.2 Pipet 50 µL of each SCV stock into 9.9 mL of 5% HCl/5% HNO_3 reagent blank. This is for digested aqueous samples.

12.17.2.3 Pipet 50 µL of each SCV stock into 9.9 mL of 0.5% HNO_3 reagent blank. This is for non-digested aqueous samples.

12.17.3 Refer to Attachment 23.12, Table 11, SCV Concentration when diluted according to instructions.

13.0 SAMPLE PREPARATION

13.1 Soil, sludge, oil and waste samples must be digested prior to analysis. Refer to TriMatrix SOP GR-01-137 for the digestion procedure.

13.2 Leachates, such as from the TCLP or SPLP extraction, must be digested. Refer to TriMatrix SOPs GR-01-119, GR-01-117, and GR-01-147 for extraction and digestion.

13.3 Water and groundwater samples for total metals must be digested. Refer to TriMatrix SOP GR-01-147 for the digestion.

14.0 CALIBRATION PROCEDURES

14.1 The Optima 3000, 3300DV and 5300DV must be calibrated for every analytical run or every 24 hours, whichever is more frequent.

14.1.1 Before analysis can begin, the instrument must be profiled using a 10 mg/L Mn solution.

14.1.1.1 Click on the tools drop down menu, then spectrometer control.

14.1.1.2 Aspirate the 10 mg/L Mn solution.

14.1.1.3 Click on "optimize".

14.1.1.4 The instrument will take several readings at different positions in the plasma path.

14.1.1.5 When readings are complete, the instrument will set the viewing position to the highest emission count.

14.1.1.6 Record the highest emission count and position setting in the maintenance logbook.

14.1.1.7 If emission readings vary by more than 30% from the recorded value due to wear or instrument maintenance, re-profile the emission output.

14.1.2 Calibration standards are prepared according to Section 12.0. The final concentrations of the working standards used to create the curve are listed in Table 5.

Note: Concentration of the lowest calibration standard must be at or below the reporting limit.

14.1.3 The mean response of a minimum of two exposures of each standard to the plasma is used in generating a calibration curve.

14.1.4 In all cases, calibration is performed using a multi-point calibration with a calculated zero intercept (not forced). The correlation coefficient must be greater than or equal to 0.995. If correlation is less than 0.995, recalibrate the instrument or postpone analysis for only out-of-control elements, until an acceptable calibration can be attained. If correlation is still less than 0.995, contact the metals laboratory supervisor for assistance.

14.2 After calibration, run an initial calibration verification

14.2.1 Analyze the secondary calibration verification (SCV). The SCV standard must be prepared from an independent (second-source) material at or near the mid-range of the calibration curve. The control window for the SCV is 90% to 110% recovery of the true value. If the percent recovery of any target analyte does not fall within the control window, reanalyze the SCV one time. If the recovery is still out of control, recalibrate the instrument and start the analysis at Section 14.2 or postpone analysis for only the out-of-control elements until an acceptable calibration can be obtained.

14.2.2 Analyze the CCB. The CCB is a reagent blank having the same acid concentration as the standards. The absolute value of the mean ICB reading must be less than the lowest reporting limit required for the analysis. If the CCB is not within the control limit, reanalyze the CCB one time. If the recovery is still out, recalibrate the instrument and start again at Section 14.2 or postpone analysis for only the out-of-control elements until an acceptable calibration can be attained.

14.2.3 Analyze the Contract Required Detection Limit (CRL). Analyze a low level standard at 2 times the standard quantitation limit. The control window is 50% to 150% recovery. If the recovery is outside of the control window, a narrative must be attached to the LIMS batch.

14.2.4 Analyze the IFA-1 solution. The IFA-1 is comprised of interfering elements only. Depending upon the analytes of interest, a second IFA-2 solution containing additional elements may also need to be analyzed. The purpose of the standard(s) is to demonstrate that, with the current interelement correction factors in place, there are no measurable spectral interferences. The QC acceptance window is 80% to 120% recovery of the true value for the elements spiked and ±2 times the reporting limit for the elements not spiked in the solution.

14.2.5 Analyze the IFB-1 solution (Interelement Correction Standard-1). The IFB-1 solution is a standard that contains four to six elements (i.e., interferents), at high concentrations with remaining elements present at trace levels. Depending upon the analytes of interest, a second IFB-2 solution containing additional elements may also need to be analyzed. The purpose of the standard(s) is to demonstrate that, with the current interelement correction factors in place, there are no measurable spectral interferences. The QC acceptance window is 80% to 120% recovery of the true value.

14.2.6 Follow analysis of the IFB standards with analysis of a Continuing Calibration Verification (CCV) standards with a concentration at the mid-point of the calibration curve. The acceptance window for this QC is 90% to 110% recovery of the true value and <5% RSD. If the CCV is not within the control limits, reanalyze the CCV one time. If the recovery is still out, recalibrate the instrument for the affected analytes and start again at Section 14.2, or postpone analysis for only the out-of-control elements until an acceptable calibration can be attained.

14.2.7 Analyze the CCB again. If the CCB is not within the control limits, reanalyze the CCB one time. If the recovery is still out, recalibrate the instrument and start again at Section 14.2, or postpone analysis for only the out-of-control elements until an acceptable calibration can be attained. All samples with an unacceptable CCB must be reanalyzed for the affected target analyte(s).

14.2.8 Analyze up to 10 samples. If the percent RSD is >20 for any result, the sample immediately prior is to be checked for a high analyte concentration. If the analyte is high, then check the replicates of the sample in question to verify the concentration is not decreasing. If the concentration is decreasing, reanalyze the sample. If the replicates are not decreasing, report the result.

14.2.9 Analyze the CCV again. If the CCV is not within the control limits, reanalyze the CCV one time. If the recovery is still out, recalibrate the instrument and start again at Section 14.2, or postpone analysis for only the out-of-control elements until an acceptable calibration can be attained. All samples with an unacceptable CCV must be reanalyzed for the affected target analyte(s).

14.2.10 Analyze the CCB again. If the CCB is not within the control limits, reanalyze the CCB one time. If the recovery is still out, recalibrate the instrument and start again at Section 14.2, or postpone analysis for only the out-of-control elements until an acceptable calibration can be attained. All samples with an unacceptable CCB must be reanalyzed for the affected target analyte(s).

14.2.11 Repeat Sections 14.2.8 through 14.2.11 until the end of the analytical run.

14.2.12 End the run with a compliant CCV and CCB analysis.

15.0 ANALYTICAL PROCEDURE

Note: The following section assumes the user is familiar with Optima 3000 and 3300DV operating software. If unfamiliar or unsure, consult the instrument manual for additional instructions.

This procedure provides adequate instruction, but is not intended to replace instrument operating manuals.

15.1 Preliminary treatment of all matrices is always necessary because of the complexity and variability of sample matrices. Digestion procedures are presented in the appropriate Sample Preparation Methods.

15.2 Turn on the instrument.

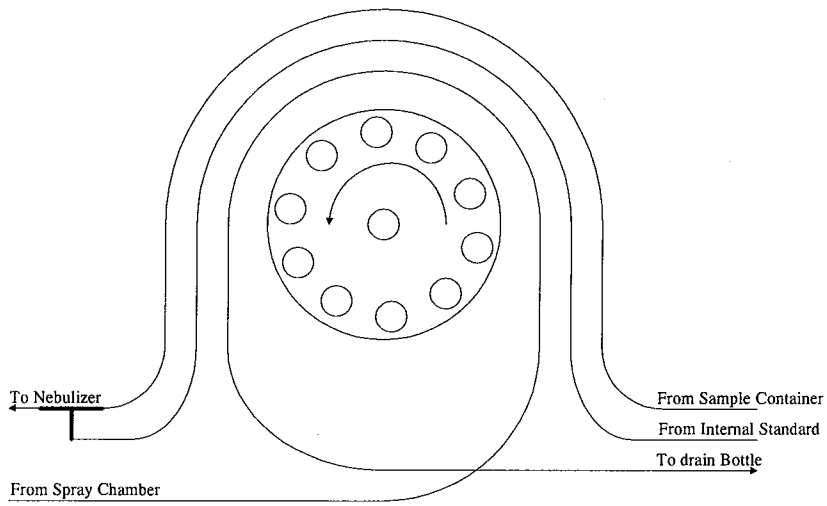
15.3 Boot the computer and ICP WinLab software.

15.4 If the instrument power or RF generator power was not on when the system was booted, an equilibration time will be required by the instrument before the plasma may be lit. The instrument software will alert the operator in this event. Wait until the instrument alerts you that it is ready before proceeding to the next step.

15.5 Change the peristaltic pump tubing. The tubing should be changed every eight hours since the tension exerted on the tubing by the pump will cause the tubing to stretch. Place the waste line on the bottom so that the pump is pumping the waste away from the ICP. Place the internal standard line in the middle so that the pump is pumping the internal standard toward the ICP. Place the sample line on the top so that the pump is pushing the sample to the ICP. Refer to Figure 1 for the correct orientation of the pump tubing. Update the instrument maintenance log to reflect that a change in the pump tubing. Currently, three types of tubing are being used:

Color	Type
Black/Black	Sample line
Orange/Green	Internal standard line
Red/Red	Waste line

Figure 1
Peristaltic Pump Setup



15.6 Recalibrate the spectrometer. The Hg recalibration helps to compensate for drift during the analysis. Although not required, this procedure will greatly increase the stability of the spectrometer, and is therefore highly recommended. Enter the spectrometer mode. Click on the Hg recalibration button. Wait until the computer states that the Hg lamp recalibration is complete. Change the Hg recal time to 2 hours.

15.7 Table 8 lists typical element file conditions for the Optima 3000. Note that any or all of the conditions listed below may change for an element if it has been established that new conditions are at least equivalent to those listed.

15.8 Refer to Attachment 23.9, Table 8, Element Conditions for the Optima 3000. Dashes indicate no Background subtraction point used for this interval.

15.9 Set up the analysis on paper using a sample identification weight sheet (ID/WT sheet). This sheet has columns where the operator enters the sample description, corresponding autosampler position number, digestion dilution (if any), and any subsequent dilutions performed on the sample. This sheet must be filled out before continuing with the procedure, as it is an integral part of the analysis. A typical starting sequence for the ID/WT sheet is given in Table 9. Consult the area supervisor if assistance is required in filling out this sheet.

15.10 Refer to Attachment 23.10, Table 9, ID/WT Sheet Setup for Instrument Calibration and Verification

15.11 Set up the analysis.

15.11.1 Import the analysis sequence from the laboratory information management system Element™ in accordance with TriMatrix GR-10-123.

15.11.2 Enter the analysis sequence.

15.11.3 Analyze samples.

15.11.4 When the analysis is complete, the autosampler will return to position "0" and wait for operator input.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The concentration of each sample is read directly from the computer printout. Dilution factors should be taken into account in the sample information file.

$$C_{Final} = C_{Instr} * DF_{Digest} * DF_{Subseq}$$

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for exporting each run to the laboratory information management system (Element™) in accordance with TriMatrix SOP GR-10-123. It is essential to perform the export correctly to place data from the instrument appropriately and accurately in Element™. Prepare data files for exporting as follows:

17.1.2 Instrument 101 (Optima 3000)

17.1.2.1 Click on "File", "Utilities" and then "Reformat".

17.1.2.2 Browse to select the dataset to be reformatted and exported.

17.1.2.3 Next, save the reformatted file to the archive directory by browsing to select "archive\101\current year\current month\current week". Be sure the reformatted file extension is ".prn".

17.1.2.4 Select the "sample" tab and click each box to show an "x".

17.1.2.5 Select the "mean" tab and click each box to show an "x".

17.1.2.6 Select the "repl" tab and click each box to change the value to the number of replicates used in the analysis.

17.1.2.7 At this point, click "save results" then close the reformatting window.

17.1.3 Instrument 116, and 311 (Optima 3300DV, 5300DV)

17.1.3.1 Click on "File", "Utilities", then "Data Manager".

17.1.3.2 Highlight the dataset to be exported then click "Export".

17.1.3.3 Click "New Design", then "Next" and then "Next" again on the two following windows without making any changes.

17.1.3.4 Change the dataset file name extension to ".prn".

17.1.3.5 Change the directory to archive\me116(311)\current year\export\current month\current week then click "next".

17.1.3.6 Highlight all available parameters and click "Add" then "Next". Repeat for the following page.

17.1.3.7 Finally, click "Export Data" and when complete, close the window.

17.2 If internal chain-of-custody is required, it is very important that the COC form be filled in correctly and returned to the COC file location.

17.3 All hardcopy generated from the analysis must be archived appropriately.

17.4 All logbooks must be filled in completely and correctly. Corrections are to be made with a lineout, not a writeover, and must be dated and initialed. Blank lines on a logbook page must be Z'd out.

18.0 QUALITY ASSURANCE

18.1 All quality control data must be maintained and available for easy reference or inspection.

18.2 Linear range studies must be performed quarterly (or more frequently if specified by project DQO's), or when there is a significant change in instrument response.

18.2.1 To perform a linear range study, calibrate the instrument as it would be calibrated for an actual analysis.

18.2.2 Run standards at high concentrations and calculate the percent recovery for each element. A good place to start is 100 mg/L.

18.2.3 If the percent recovery is acceptable (90%-110%), run a standard at a higher concentration. If the percent recovery is not acceptable, run a standard at a lower concentration.

18.2.4 The last standard concentration that had a percent recovery within the acceptance window will be designated as the linear range for that element.

18.3 IDL studies must be performed after interelement correction factors are updated. Calculate the IDL as follows:

18.3.1 Analyze seven sequential aliquots of metals reagent water. Repeat on two more non-consecutive days to obtain 21 results in 3 datasets.

18.3.2 Input these data to the IDL spreadsheet located on the laboratory intranet to calculate the IDL which is the calculated average standard deviation.

18.3.3 The spreadsheet compares the calculated IDL to the calculated method detection limit (MDL). The IDL must be less than the MDL. If not, locate the source of the problem and repeat the IDL/MDL studies successfully. Repeat only for elements that failed.

18.3.4 Consult the metals supervisor and/or the quality assurance department if repeated studies indicate the IDL is not less than the MDL.

18.4 Interelement correction factors must be updated every 6 months.

18.4.1 Interelement correction factors must be used.

18.4.2 To update or create factors, pure single-element standard solutions of interfering elements are aspirated into the instrument at levels equivalent to those found in samples. The instrument measures the intensity of all of the analytes, and the intensity of the interfering element. The ratio of false analyte intensity to interfering element intensity is called the interelement correction factor. The factors may be positive or negative depending on if the interfering element causes a false positive or false negative reading for the analyte.

18.5 Dilution is not required for samples/digestates exceeding the calibration range until 90% of instrument linearity is exceeded (Section 18.2).

18.5.1 If a project specifically requires quantitation within the calibration range, analyze a single-point standard more concentrated than the sample/digestate. If the single-point result is between 90%

and 110% of the expected concentration based on the existing calibration, sample/digestate results between the highest calibration point and the single-point standard may be quantitated without diluting.

18.5.2 The single-point standard concentration must remain equal to or less than 90% of the established linear range. The high level standard must be analyzed prior to analysis of the closing CCV/CCB.

18.5.3 Any sample/digestate concentration exceeding 90% of the linear range must be diluted.

18.6 Include a minimum of one Method Preparation Blank (BLK) per sample batch to determine if contamination or any memory effects are occurring. The BLK must be carried through all sample preparation. Results must be at or below the reporting limit.

18.7 Include a minimum of one Laboratory Fortified Blank (BS) per sample batch. The BS must be carried through the sample preparation procedure. The BS is reagent water spiked with each analyte between the low and mid-level standard except minerals, which are spiked at a higher level. The acceptance window is not to exceed 80 to 120% recovery of the prepared concentration. When historical limits are available, they may be used if within the 80 to 120% maximum. If the BS is outside the acceptance window, re-run once. If still outside the acceptance window, all samples associated with that BS must be re-prepared and re-analyzed.

18.8 Analyze one matrix spike (MS), and one matrix spike duplicate (MSD), at a frequency of at least 1 in 20 (5%). MS and MSDs are aliquots of sample to which is added a known quantity of analyte between the low and mid-level standard except minerals, which are spiked at a higher level. The percent recovery of the analyte is calculated. The percent recovery is not to exceed 75% to 125% recovery (or in-house established limits) with an RPD <20%. This measures the accuracy of the sample preparation method as well as the effect of the matrix on the analysis. The relative percent difference is calculated from the MS and MSD concentrations. The MSD checks the precision of the method.

18.8.1 Pretreated samples must be spiked before the digestion has begun. Refer to the specific digestion procedure for a discussion on spiking.

18.8.2 Samples not requiring pretreatment are spiked before they are physically loaded on the tray.

18.8.2.1 Currently, there are two 'spiking solutions' used to perform the addition of analyte to the sample. These are designated as SSW-1A and SSW-SSS-2A. These solutions are stable by themselves, but due to the high concentrations involved would not be compatible together.

18.8.2.2 Refer to Attachment 23.11, Table 10, Spiking Solutions Final Concentrations

18.8.3 Calculate the percent recovery of the MS and MSD as follows:

$$\%R = \frac{C_{Spike} - C_{Orig}}{\text{Spike Quantity}} \times 100$$

where:
%R is the percent recovery of the spike
C_{Spike} is the concentration of the spiked sample

C_{Orig} is the original concentration of the sample (the unspiked sample concentration)
Spike Quantity is the quantity of the element spiked into the sample

18.8.4 Calculate the relative percent difference between the MS and MSD as follows,

$$\%RPD = \frac{C_{MSD} - C_{MS}}{(C_{MSD} + C_{MS})/2} \times 100$$

where:

%RPD is the relative percent difference between the MS and MSD
C_{MSD} is the concentration of the MSD as read from the raw data printout
C_{MS} is the concentration of the MSD as read from the raw data printout

18.8.5 The MS must recover within 75-125% (or within laboratory established limits) of the true value. The precision between the two spikes must be ≤20%.

18.9 The following tests outlined below are performed with each batch of samples and will ensure that neither positive nor negative interferences are affecting the measurement of any of the elements or distorting the accuracy of the reported values.

18.9.1 Post-Digestion Spike (PS): The same sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. An analyte spike between the low and mid range standard is added to a portion of a prepared sample, or its dilution. The acceptance window is 80% to 120% of the true value. If the MS/MSD recovery was acceptable and the PDS was unacceptable then a serial dilution should be performed on this sample. If the MS/MSD recovery was unacceptable, and the PDS recovery was unacceptable then a matrix effect is confirmed. An alternative test method should be considered.

18.9.2 Serial Dilution: A serial dilution should be performed on the same sample as the MS/MSD. If the analyte concentration is ≥50 times the MDL, the analysis of a 1:5 dilution should agree within ±10% of the original determination. If not, then a chemical or physical interference effect should be suspected. The use of standard-addition analysis can usually compensate for this effect.

18.9.3 Duplicate (DUP) Analysis: If specified for the project, a DUP is analyzed in substitution for, or in addition to, the MSD. A DUP is a second aliquot of sample prepared and analyzed in the same way as the sample. The acceptance window for the DUP is ≤10% RPD.

18.10 Method of Standard Additions (MSA)

18.10.1 The MSA technique must be performed on all TCLP extracts that have spike recoveries less than 50% or if the concentration is within 20% of the regulatory limit. Regulatory limits are as follows:

Arsenic	5.0 mg/L
Barium	100 mg/L
Cadmium	1.0 mg/L
Chromium	5.0 mg/L

Copper	100 mg/L
Lead	5.0 mg/L
Selenium	1.0 mg/L
Silver	5.0 mg/L
Zinc	500 mg/L

18.10.2 This technique is used when interferences in the sample enhance or depress the analytical signal, resulting in a biased high or low quantitated value. The MSA technique will not correct additive interferences that cause a baseline shift.

18.10.3 The standard addition technique involves adding known amounts of standard to three aliquots of the sample solution. The sample aliquots are then quantitated as discussed in the SOP. The quantitated values of the sample and the three spikes are used to construct a linear regression curve. The curve is extrapolated to find the point where it crosses the x axis. The absolute value of this point is the analyte concentration in the sample.

18.10.4 Prepare three aliquots with spikes at 50%, 100%, and 150% of the amount found in the sample.

18.10.5 Quantitate the samples following the SOP. Using the regression program located on the computer in the metals lab, plot the absorbance vs. the concentration to construct a curve using the four results. The extrapolated x-intercept point is the concentration of the analyte. An example of a plot so obtained is shown in Attachment 23.4.

Note: For results of this technique to be valid, the following limitations must be taken into consideration:

18.10.5.1 The curve constructed must be linear, with a correlation coefficient of ≥0.995.

18.10.5.2 The chemical form of the analyte added must respond the same way as the analyte in the sample.

18.10.5.3 The interference effect must be constant over the working range of concern.

18.10.5.4 The signal must be corrected for any additive interference.

18.11 Internal Standardization must be used in all analysis to correct for instrument drift, and physical interferences. The recovery requirement for the internal standard is 80% to 120% of the true value.

18.11.1 If the internal standard recovery is low, the sample must be diluted 1:5 and reanalyzed. If the recovery is still low, a further 1:5 dilution is required. This process is repeated until satisfactory recoveries are achieved.

18.11.2 If the internal standard recovery is high, then the internal standard may naturally be present in the sample, and a different internal standard must be used.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Spike four aliquots of metals reagent water so concentration is in the lower half of the calibration range.

19.1.1.2 Digest and analyze the four spikes as samples following every step in the procedures.

19.1.1.3 Input the four results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation. Average recovery must be within blank spike control limits. Relative standard deviation must be ≤20%.

19.1.1.4 If either criterion is not met, locate and correct the source of the problem and repeat the study successfully.

19.1.1.5 Repeated failure will confirm a general problem with the procedure and/or techniques used. If this occurs, locate and correct the source of the problem, revise the procedure and/or techniques uses then repeat the study successfully.

19.1.1.6 Samples may not be analyzed by any analyst or on any instrument until a successful demonstration of capability study has been completed.

19.1.2 A Continuing Demonstration of Capability (CDC) study must be performed annually by one of the following:

19.1.2.1 By repeating the IDC study.

19.1.2.2 By using four consecutively analyzed blank spikes obtained from routine sample analysis if run exclusively by the analyst.

19.1.2.3 By successfully analyzing a performance testing sample during the course of routine sample analysis if run exclusively by the analyst.

19.1.2.4 By using the last four of seven results from an MDL study if run exclusively by the analyst. ONLY the last four results may be used.

19.1.3 Submit copies of all successful demonstrations of capability spreadsheets to the quality assurance department for data and training documentation.

19.2 A Method Detection Limit (MDL) study must be performed on every instrument using this procedure and for each digestion procedure. Refer to TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all procedures in TriMatrix Laboratory SOP number GR-15-102, *Laboratory Waste Disposal*, most recent revision, for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Method 6010C, "Inductively Coupled Plasma-Atomic Emission Spectrometry", Revision 3, February 2007

22.2 *ICP Winlab Software Guide*, Revision E, June 1997

22.3 *Optima 3000 Family Hardware Guide*, Release B, August 1997

23.0 ATTACHMENTS

23.1 Table 1, Analytical Wavelengths and Instrumental Reporting Limits for Optima 3000.

23.2 Table 1A, Analytical Wavelengths and Viewing for Optima 3300DV.

23.3 Table 2, Analyte Emission Equivalents Arising from Interference for the Optima 3000, Instrument 101.

23.4 Table 3, 100X Concentration OPT A

23.5 Table 4, 100X Concentration OPT B

23.6 Table 5, Optima Working Standard Concentrations

23.7 Table 6, IFB-1 Working Solution

23.8 Table 7, IFB -2 Working Solution

23.9 Table 8, Element Conditions for the Optima 3000

23.10 Table 9, ID/WT Sheet Setup for Instrument Calibration and Verification

23.11 Table 10, Spiking Solutions Final Concentrations

23.12 Table 11, SCV Concentration When Diluted According to Instructions

23.13 Table 12, 100X Concentration CRL 1

23.14 Table 13, 100X Concentration CRL 2

Attachment 23.1
Table 1
Analytical Wavelengths And Nominal Reporting Limits For The Optima 3000

Element	Elemental Symbol	Wavelength (nm)	Reporting Limit (ug/L)	Reporting Limit (mg/Kg)
Aluminum	Al	396.140	100	10
Antimony	Sb	206.831	100	10
Arsenic	As	188.979	100	10
Barium	Ba	455.403	10	1.0
Beryllium	Be	234.861	1	1.0
Boron	B	249.773	100	10
Cadmium	Cd	214.438	10	1.0
Calcium	Ca	315.887	500	50
Chromium	Cr	205.558	10	1.0
Cobalt	Co	230.786	10	1.0
Copper	Cu	324.754	10	1.0
Iron	Fe	238.204	10	1.0
Lead	Pb	220.353	50	5.0
Lithium	Li	670.781	8	1.0
Magnesium	Mg	279.074	500	50
Manganese	Mn	257.610	10	1.0
Molybdenum	Mo	202.031	100	10
Nickel	Ni	231.603	10	1.0
Phosphorus	P	213.618	250	25
Potassium	K	766.514	500	50
Scandium	Sc	357.253	N/A	N/A
Selenium	Se	196.026	100	10
Silicon	Si	251.611	500	50
Silver	Ag	328.065	10	1.0
Sodium	Na	588.995	500	50
Strontium	Sr	407.771	50	5.0
Thallium	Tl	190.800	100	10
Tin	Sn	189.933	1000	100
Titanium	Ti	334.941	100	10
Vanadium	V	292.402	10	1.0
Yttrium	Y	360.071	N/A	N/A
Zinc	Zn	206.200	10	1.0

Attachment 23.2
Table 1A
Analytical Wavelengths And Viewing For Optima 3300DV

Element	Elemental Symbol	Wavelength (nm)	View	Reporting Limit (ug/L)	Reporting Limit (mg/Kg)
Aluminum	Al	396.154	Radial	100	10
Antimony	Sb	217.580	Axial	100	10
Arsenic	As	188.979	Axial	100	10
Barium	Ba	455.401	Radial	10	1.0
Beryllium	Be	313.100	Axial	1	1.0
Boron	B	182.576	Axial	100	10
Cadmium	Cd	214.435	Axial	10	1.0
Calcium	Ca	315.887	Radial	500	50
Chromium	Cr	205.557	Axial	10	1.0
Cobalt	Co	228.613	Axial	10	1.0
Copper	Cu	324.754	Axial	10	1.0
Iron	Fe	238.201	Radial	10	1.0
Lead	Pb	220.351	Axial	50	5.0
Lithium	Li	670.782	Radial	8	1.0
Magnesium	Mg	279.074	Radial	500	50
Manganese	Mn	257.606	Radial	10	1.0
Molybdenum	Mo	202.030	Axial	100	10
Nickel	Ni	231.603	Axial	10	1.0
Phosphorus	P	178.222	Axial	250	25
Potassium	K	766.494	Radial	500	50
Scandium	Sc	357.253	Radial	N/A	N/A
Selenium	Se	196.027	Axial	100	10
Silicon	Si	251.609	Radial	500	50
Silver	Ag	328.067	Axial	10	1.0
Sodium	Na	588.998	Radial	500	50
Strontium	Sr	407.769	Radial	50	5.0
Sulfur	S	181.971	Axial	200	20
Thallium	Tl	190.792	Axial	100	10
Tin	Sn	189.927	Axial	1000	100
Titanium	Ti	334.939	Radial	100	10
Vanadium	V	292.399	Axial	10	1.0
Yttrium	Y	360.071	Axial	N/A	N/A
Zinc	Zn	206.198	Axial	10	1.0

Attachment 23.3
Table 2
Analyte Emission Equivalents Arising From Interference For The Optima 3000
Instrument 101

Analyte	□	Al	B	Be	Ca	Cd	Co	Cu	Fe	K	Li	Mg	Mn
Ag	328.065	0.000050	0.000010	-0.000002	-0.000186	-0.000009	-0.000026	-0.000004	-0.000841	-0.000002	---	-0.000914	0.000056
Al	396.140	N/A	0.000015	---	0.000601	0.000015	0.000002	0.000001	-0.000144	0.000005	-0.000001	0.000034	---
As	193.694	-0.000015	-0.000003	---	-0.000001	0.000026	0.000022	-0.000002	-0.000010	---	---	-0.000014	-0.000001
B	249.773	0.000017	N/A	-0.000013	0.000026	0.002253	-0.000094	---	0.002248	0.000008	0.000001	-0.000027	0.000004
Ba	455.403	0.000103	-0.000139	0.000084	0.000055	0.000177	0.008286	0.000013	-0.000429	0.000177	0.000015	0.000229	0.000014
Be	234.861	0.000260	0.000032	N/A	0.000004	0.000047	0.000153	0.000003	0.001322	0.000158	0.000005	-0.000152	-0.000008
Ca	315.887	0.000046	-0.000416	-0.000041	N/A	-0.000459	0.008221	0.000016	0.000264	-0.000099	-0.000011	0.000794	0.000019
Cd	214.438	0.000114	-0.000001	-0.000038	0.000001	N/A	0.000053	0.000022	0.000109	0.000001	---	0.000145	0.000001
Co	238.890	---	-0.000061	0.000002	-0.000005	-0.000046	N/A	---	0.001418	0.000003	---	0.000006	0.000001
Cu	324.764	0.000025	0.000061	0.000013	-0.000030	0.000041	0.000496	N/A	-0.001169	0.000014	0.000005	0.005583	0.000041
Fe	238.204	0.000037	0.000025	0.000004	0.000008	0.000009	0.000098	0.000032	N/A	0.000007	0.000001	0.000396	0.000024
K	766.514	0.000021	0.000098	0.000001	0.000010	0.000017	-0.000066	0.000012	0.000770	N/A	0.000014	0.000108	0.000040
Li	670.781	0.000018	0.000438	-0.000003	-0.000053	0.000251	0.000042	-0.000009	0.000619	0.000028	N/A	0.001026	-0.000007
Mg	279.074	0.000006	0.000005	-0.000001	-0.000026	-0.000004	-0.000183	---	0.000100	-0.000002	---	N/A	0.000053
Mn	257.610	0.000145	-0.000031	0.000001	0.000016	-0.000045	0.000053	0.000004	-0.000116	0.000011	---	0.001103	N/A
Mo	202.031	-0.000007	0.000002	---	-0.000002	---	-0.000005	0.000005	-0.000005	---	---	0.000005	---
Na	588.995	0.000530	0.002112	0.000129	0.000310	0.002348	0.002388	0.000069	-0.002495	0.000726	0.000111	0.001083	0.000116
P	213.618	0.000008	-0.000006	---	-0.000005	-0.000001	-0.000004	0.000202	-0.000018	-0.000002	---	0.000011	-0.000001
Sb	206.831	-0.000027	0.000005	---	-0.000002	-0.000005	0.000006	-0.000002	-0.000008	---	---	0.000019	-0.000002
Sc	357.253	0.000268	-0.000450	-0.000056	0.000136	-0.000875	-0.001421	-0.000010	-0.000510	-0.000126	-0.000023	0.001730	0.000001
Se	196.026	-0.000001	-0.000010	---	0.000001	0.000002	-0.000049	---	-0.000022	---	---	-0.000006	---
Si	251.611	0.000554	-0.000130	-0.000009	0.000319	-0.000278	-0.000358	0.000028	-0.000091	-0.000055	-0.000006	0.009041	0.000015
Sr	407.771	0.000007	0.000227	0.000002	0.006871	0.000242	-0.001019	0.000002	-0.000223	0.000010	0.000292	-0.000004	0.000002
Ti	334.941	0.000092	0.000081	0.000006	-0.000424	0.000159	0.000752	0.000102	0.000262	0.000028	0.000003	0.001648	0.000007
V	292.402	-0.000012	0.000028	-0.000013	0.000002	0.000028	0.000019	0.000001	0.000401	0.000003	0.000001	0.000052	0.000004
Pb	220.353	0.000002	-0.000003	-0.000006	0.000003	-0.000008	0.000072	0.000005	-0.000011	---	---	-0.000062	-0.000001
Ni	231.603	-0.000011	0.000007	---	---	0.000023	0.001143	-0.000002	-0.000007	---	---	-0.000046	---
Cr	205.558	0.000005	0.000007	-0.000535	0.000001	0.000010	0.000020	-0.000001	0.000010	---	---	-0.000014	-0.000003
Zn	213.858	0.000035	0.000020	-0.000005	0.001138	0.000076	0.000062	0.000494	0.000146	0.000008	0.000002	0.000049	0.000006
Sn	189.930	-0.000005	0.000003	---	0.000029	-0.000005	0.000009	-0.000001	-0.000020	0.000001	---	0.002130	-0.000004
Tl	190.794	0.000066	-0.000131	-0.000001	0.000026	-0.000210	-0.000102	-0.000001	-0.000557	-0.000030	-0.000004	-0.000844	0.000007

Attachment 23.3
Table 2 Continued
Analyte Emission Equivalents Arising From Interference For The Optima 3000
Instrument 101

Analyte	□	Mo	P	Sb	Sc	Se	Ti	V	Pb	Ni	Cr	Ag	Tl
Ag	328.065	0.001737	0.000341	0.000406	0.000030	0.001644	0.000045	0.000229	0.003710	-0.000024	-0.000049	-0.000243	0.000172
Al	396.140	0.050458	0.000335	0.000426	-0.000006	0.000179	0.000017	0.000036	0.000070	-0.000520	0.000060	-0.000001	-0.000035
As	193.694	0.000032	-0.000084	-0.000118	-0.000004	0.000177	---	0.000015	0.000062	0.000026	0.000140	0.000002	---
B	249.773	-0.000677	0.000744	0.000266	-0.000004	0.044662	0.000010	-0.000148	0.016232	0.000263	-0.000138	0.000372	0.047492
Ba	455.403	0.004130	0.019290	0.007999	0.000193	0.001338	-0.000023	0.000194	0.003324	0.000472	0.000780	0.000004	0.000881
Be	234.861	-0.005137	0.005041	0.001900	-0.000008	0.001585	0.000007	-0.000001	0.000537	0.000059	-0.001748	0.000006	0.000281
Ca	315.887	0.010015	-0.005816	-0.004457	0.000089	-0.018792	0.000036	-0.003852	-0.005777	0.000046	-0.001528	-0.000099	-0.003067
Cd	214.438	-0.000123	0.000197	0.000387	-0.000004	0.000335	---	-0.000051	0.000228	0.000817	-0.000039	0.000021	0.000032
Co	238.890	0.000090	0.000323	-0.000024	0.000004	0.000070	---	0.000246	0.001663	-0.000037	-0.000054	-0.000003	0.000043
Cu	324.764	0.022074	0.001118	0.000404	0.000076	0.003799	-0.000125	0.000038	0.000981	0.005341	0.000108	0.000014	0.000200
Fe	238.204	0.000118	0.001063	0.001195	0.000007	0.001845	0.000165	-0.001028	0.000438	0.000094	0.004045	0.000007	0.000276
K	766.514	0.002393	0.011369	0.002821	0.000143	0.006884	0.000014	0.000286	0.000204	0.000179	-0.000065	0.000014	0.000156
Li	670.781	-0.000933	0.002374	0.010223	-0.000068	0.006715	-0.000005	0.000108	0.001937	-0.000191	-0.000305	0.000041	0.001957
Mg	279.074	0.000157	-0.000098	0.000029	-0.000004	-0.000059	0.000028	0.000041	-0.000072	-0.000038	-0.000412	-0.000003	-0.000070
Mn	257.610	-0.000385	-0.000633	-0.000299	0.000002	-0.000978	0.000047	-0.000066	-0.000433	0.001016	0.002430	-0.000003	-0.000190
Mo	202.031	N/A	0.000052	0.000011	0.000001	-0.000038	0.000001	0.000004	0.000025	0.000074	0.001457	---	0.000014
Na	588.995	0.003140	0.053565	0.044226	0.000513	0.101270	0.000033	0.000344	0.027038	0.001390	0.002427	0.000416	0.014623
P	213.618	0.001570	N/A	-0.000099	0.000003	-0.000327	-0.000001	0.000002	-0.000166	0.000112	0.000037	-0.000002	-0.000048
Sb	206.831	0.001094	-0.000108	N/A	-0.000070	0.000026	---	0.000001	-0.000033	0.000093	0.001717	---	0.000061
Sc	357.253	0.003392	-0.015891	-0.009545	N/A	-0.030062	-0.000006	0.000634	0.009755	-0.002416	-0.000594	-0.000137	-0.004557
Se	196.026	0.000015	0.000109	0.000032	-0.000002	N/A	---	---	0.000018	0.000004	0.000014	---	0.000009
Si	251.611	0.017841	-0.007392	-0.004843	0.000492	-0.010804	0.000385	0.000471	-0.004126	0.000438	0.000570	-0.000045	-0.001029
Sr	407.771	0.001288	0.002436	0.096894	0.000113	0.007340	-0.000041	0.000046	0.003409	0.000069	0.002546	0.000037	0.004749
Ti	334.941	0.001154	0.002839	0.002701	0.000386	0.005029	N/A	0.002462	0.001298	0.000036	0.011714	0.000027	0.000660
V	292.402	-0.016818	0.000583	0.000402	0.000101	0.001176	0.000049	N/A	0.000325	0.000067	0.001418	0.000003	0.000099
Pb	220.353	-0.001211	-0.000046	-0.000153	0.000007	0.001118	-0.000008	-0.000010	N/A	0.000046	-0.000004	---	-0.000034
Ni	231.603	-0.000540	0.000047	0.001160	0.000003	-0.000118	0.000003	-0.000009	0.000001	N/A	-0.000047	---	0.002266
Cr	205.558	0.000666	0.000083	0.000052	0.000002	0.000532	0.000014	0.000012	0.000068	0.000925	N/A	0.000002	0.000062
Zn	213.858	-0.000080	0.001070	0.000971	0.000004	0.003067	0.000006	0.000086	0.000838	0.010323	0.000151	0.000017	0.000464
Sn	189.930	-0.000047	0.000181	0.000122	0.000005	0.000463	-0.000010	0.000002	0.000086	-0.000099	-0.000059	0.000002	0.000278
Tl	190.794	0.000124	-0.003174	-0.002041	-0.000012	-0.007422	0.000038	0.000352	-0.002062	-0.000201	0.000172	-0.000029	N/A

Attachment 23.4
Table 3
100x Concentration OPT A

Element	Concentration (mg/L)
Ca	1000
Mg	1000
Na	1000
K	1000
As	500
Se	500
P	500
Al	500
Tl	500
V	100
Ba	100
Be	10
B	100
Cu	100
Fe	100
Li	100
Zn	100
Cd	100
Cr	100
Co	100
Pb	100
Mn	100
Ni	100
Sr	100
Ce	100

Attachment 23.5
Table 4
100x Concentration OPT B

Element	Concentration (mg/L)
Sb	500
Sn	500
Si	500
Mo	100
Ti	100

Attachment 23.6
Table 5
Optima Working Standard Concentrations (mg/L)

Element	Standard 4	Standard 3	Standard 2	Standard 1
Ca	10	5	1.0	0.5
Mg	10	5	1.0	0.5
Na	10	5	1.0	0.5
K	10	5	1.0	0.1
S	5	2.5	0.5	0.1
As	5	2.5	0.5	0.10
Se	5	2.5	0.5	0.1
P	5	2.5		0.25
Al	5	2.5	0.5	0.05
Tl	5	2.5	0.5	0.1
V	1	0.5	0.1	0.01
Ba	1	0.5	0.1	0.01
Be	0.1	0.05	0.01	0.001
B	1	0.5	0.1	
Cu	1	0.5	0.1	0.01
Fe	1	0.5	0.1	0.01
Li	1	0.5	0.1	0.008
Zn	1	0.5	0.1	0.01
Cd	1	0.5	0.1	0.005
Cr	1	0.5	0.1	0.01
Co	1	0.5	0.1	0.01
Pb	1	0.5	0.1	0.05
Mn	1	0.5	0.1	0.01
Ni	1	0.5	0.1	0.01
Sr	1	0.5	0.1	0.05
Ag	1	0.5	0.1	0.005
Sb	5	2.5	0.5	0.1
Sn	5	2.5	0.5	
Si	5	2.5	0.5	0.1
Mo	1	0.5	0.1	
Ti	1	0.5	0.1	

Attachment 23.7
Table 6
IFB-1 Working Solution

Element	Concentration (mg/L)
Al	100
Ba	0.5
Be	0.5
Ca	100
Cd	1.0
Co	0.5
Cr	0.5
Cu	0.5
Fe	40
Mg	100
Mn	0.5
Ni	1.0
Pb	1.0
V	0.5
Zn	1.0

Attachment 23.8
Table 7
IFB-2 Working Solution

Element	Concentration (mg/L)
Al	1.0
As	1.0
B	1.0
Ca	5.0
Cr	20
Cu	20
Mg	5.0
Mn	20
Mo	1.0
Na	5.0
Ni	20
Sb	1.0
Se	1.0
Ti	20
Tl	1
V	20

Attachment 23.9
Table 8
Element Conditions For The Optima 3000

Analyte	λ (nm)	Peak Window (nm)	Lower BGC (nm)	Upper BGC (nm)	Viewing Height (mm)	Internal Standard	Standard Concentration	Units	Subarray Number
Ag	328.065	0.0302	0.020	---	12	Sc	1000	ug/L	147
Al	396.140	0.0361	0.048	0.060	12	Sc	5030	ug/L	236
As	188.979	0.0177	0.012	0.012	12	Sc	5050	ug/L	8
B	249.773	0.0230	---	0.031	12	Sc	1005	ug/L	82
Ba	455.403	0.0843	---	0.169	12	Sc	999	ug/L	179
Be	234.861	0.0072	---	0.036	12	Sc	1001	ug/L	74
Ca	315.887	0.0288	0.019	---	12	Sc	10.060	mg/L	137
Cd	214.438	0.0196	0.046	0.020	12	Sc	996	ug/L	40
Co	230.786	0.0220	---	0.022	12	Sc	1000	ug/L	66
Cr	205.558	0.0189	0.019	0.019	12	Sc	1000	ug/L	20
Cu	324.754	0.0396	0.030	0.030	12	Sc	1004	ug/L	153
Fe	238.204	0.0217	---	0.036	12	Sc	1004	ug/L	93
K	766.514	0.1395	0.116	0.093	12	Sc	9.959	mg/L	125
Li	670.781	0.1225	0.061	0.082	12	Sc	999	ug/L	138
Mg	279.074	0.0256	---	0.017	12	Sc	10.030	mg/L	101
Mn	257.610	0.0235	---	0.023	12	Sc	999	ug/L	91
Mo	202.031	0.0186	0.037	0.012	12	Sc	1004.5	ug/L	3
Na	588.995	0.1093	0.055	0.036	12	Sc	10.000	mg/L	148
Ni	231.603	0.0213	0.028	0.021	12	Sc	1000	ug/L	52
P	213.618	0.0196	0.033	0.013	12	Sc	5014	ug/L	38
Pb	220.353	0.0202	0.027	0.020	12	Sc	992	ug/L	54
Sb	206.831	0.0252	0.025	---	12	Sc	4987	ug/L	28
Sc	357.253	0.0326	---	0.043	12	N/A	100	%	186
Se	196.026	0.0179	0.012	0.012	12	Sc	5004.5	ug/L	15
Si	251.611	0.0230	---	0.023	12	Sc	4990	ug/L	89
Sn	189.933	0.0174	0.012	0.023	12	Sc	4992	ug/L	4
Sr	407.771	0.0749	0.037	0.050	12	Sc	996.5	ug/L	216
Ti	334.941	0.0306	---	0.041	12	Sc	1000	ug/L	169
Tl	190.800	0.0174	0.017	0.023	12	Sc	1000	ug/L	14
V	292.402	0.0270	---	0.027	12	Sc	1006	ug/L	113
Zn	206.200	0.0196	0.026	0.026	12	Sc	999	ug/L	39

Attachment 23.10
Table 9
ID/WT Sheet Setup For Instrument Calibration And Verification

Sequence Number	Autosampler Position	Sample ID	Solution Name
1	1	is_init	Blank
2	1	Cal. Blank	Blank
3	2	Std. 1	Standard 1
4	3	Std. 2	Standard 2
5	4	Std. 3	Standard 3
6	5	Std. 4	Standard 4
7	9	SCV	SCV
8	1	ICB	ICB
9	10	CRL	CRL
10	11	IFA-1	IFA-1
11	12	IFA-2	IFA-2
12	13	IFB-1	IFB-1
13	14	IFB-2	IFB-2
14	4	CCV	Standard 3
15	1	CCB	Blank

Attachment 23.11
Table 10
Spiking Solutions Final Concentrations

Element	Aqueous Samples (ug/L)	Solid Samples (ug/L)
Aluminum	1250	1250
Antimony	1250	1250
Arsenic	1250	1250
Barium	250	250
Beryllium	25	25
Boron	250	250
Cadmium	250	250
Calcium	12500	12500
Chromium	250	250
Cobalt	250	250
Copper	250	250
Iron	250	250
Lead	250	250
Lithium	250	250
Magnesium	12500	12500
Manganese	250	250
Molybdenum	250	250
Nickel	250	250
Phosphorus	1250	1250
Potassium	12500	12500
Selenium	1250	1250
Silicon	1250	1250
Silver	250	250
Sodium	12500	12500
Strontium	1250	1250
Sulfur	2500	1250
Thallium	1250	1250
Tin	1250	1250
Titanium	250	250
Vanadium	250	250
Zinc	250	250

Attachment 23.12
Table 11
SCV Concentration When Diluted According To Instructions

Element	SCV (ug/L)
Aluminum	2500
Antimony	2500
Arsenic	2500
Barium	500
Beryllium	50
Boron	500
Cadmium	500
Calcium	5000
Chromium	500
Cobalt	500
Copper	500
Iron	500
Lead	500
Lithium	500
Magnesium	5000
Manganese	500
Molybdenum	500
Nickel	500
Phosphorus	2500
Potassium	5000
Selenium	2500
Silicon	2500
Silver	500
Sodium	5000
Strontium	500
Thallium	2500
Tin	2500
Titanium	500
Vanadium	500
Zinc	500

Attachment 23.13
Table 12
100x Concentration CRL 1

Element	Concentration (mg/L)
Ca	100
Mg	100
Na	100
K	20
As	20
Se	20
P	50
Al	10
Tl	20
V	2
Ba	2
Be	0.2
B	20
Cu	2
Fe	2
Li	1.6
Zn	2
Cd	1
Cr	2
Co	2
Pb	10
Mn	2
Ni	2
Sr	10
Ag	1

Attachment 23.14
Table 13
100x Concentration CRL 2

Element	Concentration (mg/L)
Sb	20
Sn	200
Si	20
Mo	20
Ti	20

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Mercury by semi-Automated Cold Vapor Atomic Absorption

EPA Method 245.1
SW-846 Method 7470A
SW-846 Method 7471A

APPROVALS:

Area Supervisor: _____ Date: 1-19-09
Denise S. Coffey

QA Officer: _____ Date: 1-15-09
Tom C. Boocher

Operations Manager: _____ Date: 1/19/09
Jeff P. Glaser

Procedure Number: GR-01-123
Revision Number: 5.6

Date Initiated: 5/6/93
Effective Date: 2/1/09

Date Revised: 1/15/09
Pages Revised: All

By: David W. Johnson
Total Number of Pages: 21

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure includes analysis for total mercury (inorganic/organic) in mobility extracts, aqueous wastes, groundwaters, drinking waters, surface waters, saline waters, wastewaters, domestic industrial wastes, soils, wastes, sludges and oils. All samples must undergo a preliminary digestion.

1.2 The typical reporting limit is 0.2 ug/L mercury for aqueous and 0.10 mg/kg for solid sample matrices.

2.0 PRINCIPAL METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"

2.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994

2.3 *Test Method for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision I, September 1994, Method 7471A, *"Mercury in Solid and Semi Solid Waste* (Manual Cold-Vapor Technique)"

3.0 SUMMARY OF PROCEDURE

3.1 Before analysis, all analytical and quality control samples must be pretreated according to the references discussed in Section 5.0. These digestions reduce organo-mercury compounds to inorganic mercury. Inorganic mercury is then converted to mercury in the Hg^{2+} state. All Hg^{2+} with an HCl carrier enters a gas liquid separatory where it is mixed with stannous chloride to form elemental mercury vapor (Hg^0) according to the following equation:

$$Hg^{2+} + Sn^{2+} \rightarrow Hg^0 + Sn^{4+}$$

3.2 The mixture then flows into a liquid-gas separator where argon is introduced to carry the mercury vapor through a drying tube. The dried mercury vapor then enters one path of a heated double-path optical cell that has been optimized for fast response time. A mercury source powered by a constant current power supply delivers a stable source of absorbance at 253.7 nm. Absorbance by the mercury vapor is measured using a solid-state detector with a wide dynamic range. The resulting signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions.

3.3 Absorbance of standards is plotted against known concentrations to build a calibration curve. Absorbance of unknown samples are read against the calibration curve, and regressed to concentration in ug/L. The mercury vapor is vented up a hood to minimize contamination of other samples.

4.0 PARAMETER OR COMPOUND LIST

4.1 Mercury

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-01-140, *Preparation Procedure for Mercury in Water, Wastewater, and Liquid Wastes,* latest revision

5.3 TriMatrix SOP GR-01-139, *Preparation Procedure for Mercury in Soils, Wastes, and Oils,* latest revision

5.4 TriMatrix SOP GR-10-106, Inorganic *and Metals Laboratories Corrective Actions,* latest revision.

5.5 TriMatrix SOP GR-10-123, *Element Data Transfer and Review*, latest revision

5.6 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision

5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

5.8 TriMatrix SOP GR-09-128, Homogenization, Grinding and Drying of Solid Samples, latest revision

5.9 *PSA 10.045 Millenium Merlin/Galahad System User Manual,* Issue Number 1.1, Issue Date July 10, 1999

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Soil samples often contain mercury at low levels. When soils are mixed and ground, there exists the possibility that liquid samples may become contaminated by dust from the air. Therefore, preparation areas for mercury must be kept free of dust and dirt.

Note: Mixing and grinding samples must be done in a fume hood for safety.

6.2 If a mercury thermometer is broken anywhere in the laboratory, notify the metals laboratory so precautionary measures against contamination can be taken.

6.3 Mercury may be lost if the digestion temperature goes above 95° C. The digestion temperature must be monitored and recorded with a digital thermometer on the digestion benchsheet.

6.4 Reagents used may become contaminated over time. If contamination is suspected, the reagent must be discarded following proper laboratory disposal guidelines and fresh reagent made.

6.5 Potassium permanganate is added to eliminate possible interference from sulfide as sodium sulfide.

6.6 Copper interferes at concentrations above 10 mg/L.

6.7 Samples high in chlorides and volatile organic materials require additional permanganate.

Note: The maximum permanganate added to a sample in the digestion batch must be added to all associated quality control samples.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix laboratory safety manual and chemical hygiene plan.

7.2 Concentrated acids are used. Disposable gloves, safety glasses and a laboratory coat must be worn at all times when handling concentrated acids. Gloves, safety glasses and laboratory coats must also be worn when handling digested samples or performing any work within the metals laboratory. Refer to the MSDS located on the library drive for information on any chemical used.

7.3 Make sure the venting system is hooked up and running. Do not operate the instrument if it is not properly ventilated.

7.4 Mercury lamps emit UV radiation. Avoid looking directly at the lamp without some type of strong UV protection. Failure to follow this policy may cause very serious and immediate damage to the retina of the eye.

7.5 Mercury exists in many forms and is toxic in a variety of ways. Mercury vapor is toxic if inhaled. Mercury compounds can also be absorbed through the skin. Wear disposable gloves at all times. Elemental Hg^0 and Hg^{2+} are toxic if ingested. Always wash hands after handling mercury standards, samples or reagents, and when leaving the laboratory.

7.6 No food or drink is allowed in the metals laboratory. Food and drink may be consumed in the laboratory office but not stored there.

7.7 Wash hands before starting work. Chemicals may be present on the skin that can interfere with mercury analysis. Wash hands before leaving the laboratory. Chemicals and acids may be on the skin that could eventually be ingested or passed on to a third party through casual contact.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples should be acidified at the time of collection to a pH of <2. If the pH is not <2 when received by the laboratory, lower to pH <2 during the sample receipt process. A minimum of 24 hours must elapse after pH adjustment by the laboratory before performing the sample digestion.

8.2 Store non-aqueous and solid samples at 4 ±2° C.

8.3 The maximum holding time for all mercury samples is 28 days from the collection date. Analyze non-aqueous samples as soon as possible within that time.

8.4 All samples must be subjected to digestion before analysis.

8.5 The minimum collected aqueous sample volume required is 100 mL. If quality control is also required, collect at least 300 mL. The minimum sample size for solid samples is 10 g.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Laboratory analytical balance capable of weighing 0.0001 g

9.2 Laboratory balance capable of weighing 0.1 g

9.3 Macropipettor capable of delivering 50 - 1000 µL

9.4 Class A volumetric flasks, various volumes

9.5 Class A reusable pipettes, various volumes

9.6 Macropipettes capable of delivering 5 - 10 mL of solution

9.7 Wooden laboratory spatulas

9.8 Calibrated digital thermometer capable of reading to 100° C. Mercury thermometers must never be used in this procedure.

9.9 PSA low-level absorbance mercury analyzer, Millenium system, model 10.045

9.10 Computer running Avalon software. An IBM compatible computer running Windows NT 4.0 service pack 5.0. The software version may be updated without notice if it performs at least as well as the older version.

9.11 Filter, 0.45 µm glass fiber.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Check the calibration on autopipettors daily. Remove from service and send out for recertification when daily checks indicate unacceptable accuracy or performance. Perform an annual inspection to determine overall condition.

10.2 Verify balance calibration daily. Remove from service and call for repair when daily checks indicate unacceptable accuracy or performance. Recertify annually through a contracted service representative.

10.3 Inspect the waste bottle under the instrument before starting. If the bottle is full, properly dispose of the liquid.

10.4 Under normal conditions, the PSA requires very little maintenance. However, attention to the maintenance schedule below must be followed:

10.4.1 Daily:

10.4.1.1 Before shutting down, flush all tubing and the sample valve with laboratory reagent water. Finally, flush with air.

10.4.1.2 It may necessary to replace all tubing. However, flushing of the sample valve must still take place.

10.4.1.3 Replace the pump tubing if required. When in doubt, replace it.

10.4.1.4 Ensure that glassware is clean. When in doubt, clean it.

10.4.1.5 Clean all outer surfaces of the instrument.

10.4.1.6 Also after shutting down, release any tension on the pump tubing.

10.4.2 Weekly:

10.4.2.1 Perform daily maintenance activities.

10.4.2.2 Replace all pump tubing if not already replaced as part of the daily maintenance.

10.4.3 Monthly:

10.4.3.1 Perform daily and weekly maintenance activities.

10.4.3.2 Replace all pump tubing if not already replaced as part of daily/weekly maintenance.

10.4.3.3 Check for discoloration in the Perma-pure drying system and replace as necessary.

10.4.4 Six Months:

10.4.4.1 Perform daily, weekly and monthly maintenance.

10.4.4.2 Replace the inner membrane of the Perma-pure drying system if needed.

10.4.5 Yearly:

10.4.5.1 Perform daily, weekly, monthly and six month maintenance.

10.4.5.2 Change the inner membrane of the Perma-pure drying system if needed.

10.4.5.3 Replace the activated charcoal in the exhaust line.

10.4.6 Cleaning:

10.4.6.1 Before undertaking any cleaning, disconnect the main supply cable from the instrument. Cleaning is ONLY recommended for the outer case.

10.4.6.2 Clean with a damp cloth or sponge which has been previously immersed in warm soapy water. Alternatively, a suitable laboratory cleaning agent may be used. Avoid copious amounts of liquid coming into contact with the instrument.

10.4.6.3 Finish off by wiping down with a dry lint-free cloth or sponge.

10.4.7 Wipe acid spills up with a suitable neutralizing agent IMMEDIATELY. However, be careful to avoid a copious volume of liquid coming into contact with the instrument.

11.0 CHEMICALS AND REAGENTS

11.1 Acids used in standards preparation and for samples must be trace metal grade or better.

11.1.1 Concentrated hydrochloric acid, trace metal grade

11.1.2 Concentrated nitric acid, trace metal grade

11.2 ASTM Type II water

11.2.1 Deionized water is fed into an all-glass distillation unit. The resulting distillate is immediately placed into a plastic container. This is the only water acceptable for mercury analysis.

11.3 Argon gas supply

11.3.1 Welding grade or better. This is plumbed from a liquid argon tank located outside the building. Argon is used as the carrier gas during analysis.

11.4 Stannous chloride, 2% solution in 10% hydrochloric Acid.

11.4.1 Place 20 g stannous chloride into a clean 1 L polyethylene bottle. Add 100 mL concentrated HCl and swirl until the SnCl is dissolved. Add 880 mL ASTM Type II water then cap and mix.

11.4.2 Create a new standard number in Element™ when the stannous chloride lot changes.

12.0 STANDARDS PREPARATION

12.1 Refer to TriMatrix SOP GR-01-139 and SOP GR-01-140, for the preparation of mercury standards.

13.0 SAMPLE PREPARATION

13.1 Refer to TriMatrix SOP GR-01-139 and SOP GR-01-140 for the preparation of samples by matrix.

14.0 CALIBRATION PROCEDURES

14.1 A blank and five standards comprise the initial calibration curve. Refer to TriMatrix SOP GR-01-139 and SOP GR-01-140 for the preparation of calibration standards.

14.2 Prepare a digested calibration curve the same day as digestates and use for quantitation.

15.0 ANALYTICAL PROCEDURE

15.1 Analysts must be familiar with the software's operation by reading the software manual and through documented training from an experienced analyst before proceeding.

15.2 After running the calibration curve, analyze a second-source calibration verification (SCV) as a sample. The SCV is prepared from a second-source purchased standard and prepared at 3.0 ug/L. SCV recovery must be within 95 – 105% of the expected value for method reference 245.1 or within 90 – 110% for method references 7470A and 7471A.

15.2.1 If the SCV fails recovery, repeat the analysis.

15.2.2 If the second analysis fails, initiate corrective action:

15.2.2.1 Adjust the gas flow if needed.

15.2.2.2 Replace tubing if needed.

15.2.2.3 Check for other mechanical problems to verify the instrument is operating correctly.

15.2.3.4 Remake and redigest the calibration standards if necessary.

15.2.3.5 Remake the SCV standard if necessary.

15.2.3 After resolving the problem, recalibrate the instrument and run the SCV successfully.

15.2.4 Digestions may not be analyzed without an acceptable SCV.

15.3 Analyze a calibration blank (CCB) after the SCV. The mercury concentration found in the CCB must not be greater than the absolute value of the reporting limit to be acceptable.

15.4.1 If the CCB is higher than the reporting limit, reanalyze one time.

15.4.2 If the second analysis fails, initiate corrective action.

15.4.2.1 Locate the source of the contamination.

15.4.2.2 Correct the contamination problem.

15.4.2.3 Repeat all associated sample digestions associated with the contamination problem and/or narrate the contamination.

15.5 Analyze a contract-required limit (CRL) standard at the beginning of each run.

15.5.1 A CRL is reanalysis of the digested 0.2 ug/L standard from the initial calibration curve as a sample.

15.5.2 CRL recovery must fall within 50 – 150% of the expected value. If the CRL result is above the calculated MDL value but outside the control limits, narrate in the analytical batch as follows:

15.5.2.1 The CRL recovery for this analyte was outside laboratory control limits. However, the only requirement is that the analyte be detected. No qualifications are necessary.

15.6 After analysis of the CRL, analyze a continuing calibration verification (CCV) standard. The CCV is the midpoint calibration standard analyzed as a sample. CCV recovery must be within 90 – 110% of the expected value.

15.6.1 If the CCV fails, reanalyze one time.

15.6.2 If the CCV still fails, stop the analysis and initiate corrective action.

15.6.2.1 Check to make sure the instrument is operating correctly.

15.6.2.2 Recalibrate.

15.6.2.3 Reanalyze all digestates since the last acceptable CCV.

15.7 Analyze a continuing calibration blank (CCB). The mercury concentration found in the CCB must not be greater than the absolute value of the reporting limit to be acceptable.

15.7.1 If the CCB fails, reanalyze one time.

15.7.2 If the second analysis fails, initiate corrective action:

15.7.2.1 If a high level sample is carrying over, analyze the CCB until the system is clean then reanalyze all digestates run since the last acceptable CCB.

15.7.2.2 If no apparent cause can be found, recalibrate then reanalyze all digestates run since the last acceptable CCB.

15.8 After the initial quality control has passed, analyze up to 10 samples (including the high and low concentration blank spikes) then repeat analysis of the CCV and CCB. Repeat, until all samples are analyzed.

Note: Shake all digestates before analysis. Solid matrix digestates must be filtered through a 0.45 µm filter before analysis.

15.9 Check all raw data to make sure each result is within the calibration range. If any result above the calibration range, mark the sample number and estimate a dilution factor. Make the dilution using a digestion CCB that matches the sample reagent matrix then analyze within the calibration range.

15.9 Verify the calibration after all samples have been analyzed as follows:

15.9.1 Analyze a final CCV successfully.

15.9.2 Analyze a final CCB successfully, which concludes the run.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Sample concentration is read directly from the computer printout in ug/L. Dilution factors must be taken into account in the ID/WT file and the sample information file.

16.1.1 Quantitate water, wastewater and potable water samples as follows:

$$C_{Final} = C_{Instr} \times DF_{Subseq}$$

where:

C_{Final} = The final reported concentration in ug/L
C_{Instr} = The concentration as read from the instrument printout in ug/L
DF_{Subseq} = Any dilution ratio subsequent to the digestion dilution

16.1.2 Quantitate TCLP and SPLP leachates as follows:

$$C_{Final} = \left(\frac{C_{Inst} \text{ X } DF_{subsequent}}{1000}\right)$$

Where:

C_{Final} = The final reported concentration in mg/L
C_{Inst} = The concentration as read from the instrument printout in ug/L
$DF_{subsequent}$ = Any dilution ratio subsequent to the digestion dilution
1000 = Conversion from ug/L to mg/L

16.1.3 Quantitate solid samples as follows:

$$C_{Final} = (\frac{C_{Inst} \text{ X } DF_{subsequent}}{1000})$$

Where:

C_{Final} = The final reported concentration in mg/kg
C_{Inst} = The concentration as read from the instrument printout in ug/kg
$DF_{subsequent}$ = Any dilution ratio subsequent to the digestion dilution
1000 = Conversion from ug/kg to mg/kg

16.2 Duplicate measurements are taken and recorded for each sample. The average of these two readings is reported as the final result.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for correctly filling in, handing in and filing all associated paperwork. It is essential to perform these tasks for the laboratory to provide clients with defensible data.

17.2 LIMS Reporting

17.2.1 When finished running a sample set, all data must be uploaded to Element™, the Laboratory Information Management System (LIMS) in accordance with TriMatrix SOP GR-10-123.

17.2.1.1 To export a data set from the instrument for LIMS uploading with DataTool, click on the red arrow.

17.2.1.2 Choose "comma" and "browse" then select the "groups\lab.me\export data\216" folder and click "OK".

17.2.1.3 Add a backslash (\) and type the data set filename (include ".res" on the end). For example, to export dataset y17wta2, the path appears as "groups\lab_me\exportdata\216\y17wta2.res".

17.2.1.4 Click "OK" to export the data.

17.2.2 If an internal chain-of-custody is required, it is very important that COC forms be filled in correctly and returned to the COC file location. Refer to Attachment 23.5 for an example COC form.

17.3 Laboratory Required Paperwork

17.3.1 Data backup logbook and maintenance logbooks must be filled in completely and correctly. All corrections must be made in indelible ink. **Corrections must be made with a single lineout, which is dated and initialed. Writeovers are NOT acceptable.** Erroneous results must remain legible. A new result is placed near the incorrect result. Blank lines in logbooks must be Z'd out.

18.0 QUALITY ASSURANCE

18.1 All quality control data must be maintained and available for easy reference or inspection. In addition to quality control already discussed, the following requirements apply. Corrective action must be taken for unacceptable quality control results based on instructions given in this procedure, and in TriMatrix SOP GR-10-106.

18.2 Dilute and reanalyze all digestates having concentrations greater than the highest standard on the analytical curve.

18.3 For aqueous samples, include a minimum of one digestion blank (BLK), one high blank spike (BS) and one low blank spike (BS) with each batch of up to twenty samples. For solid samples, include a minimum of one digestion blank (BLK) and one BS with each batch of up to twenty samples. The BLK monitors contamination and carryover while the BS monitors digestion efficiency based on recovery. The BLK and BS must be carried through every step in the digestion and filtration procedure.

18.3.1 A BLK is an aliquot of ASTM Type II water digested as a sample. Any mercury found in the BLK must be at a concentration less than the reporting limit to be acceptable.

18.3.2 A BS is the BLK spiked at a high and low concentration.

18.3.2.1 Calculate BS recovery as follows:

$$\text{Percent Recovery} = \frac{C_{Spike}}{\text{Spike Quantity}} \times 100$$

where:

C_{spike} = The analyzed concentration of the BS after spiking in ug/L or mg/kg

Spike Quantity = The concentration spiked into the BS in ug/L or mg/kg

18.3.2.2 Recovery of the high concentration BS must be within 85 – 115% of the expected value for aqueous samples. Statistical acceptance limits are used for solid samples. Recovery of the low concentration BS must be within 70 – 130% of the expected value. The low concentration BS is for internal use and is not reported.

18.4 Matrix spike and matrix spike duplicates are a sample spiked with a known mercury concentration. Prepare and analyze one matrix spike (MS) and one matrix spike duplicate (MSD) with each batch of up to twenty samples. The matrix spike monitors digestion efficiency in the specific sample matrix. A matrix spike duplicate is a replicate of the matrix spike using the same sample and monitors precision within the sample matrix.

18.4.1 Calculate matrix spike recovery as follows:

$$\text{Percent Recovery} = \frac{C_{Spike} - C_{Orig}}{\text{Spike Quantity}} \times 100$$

where:
C_{spike} = The quantitated MS concentration in ug/L or mg/kg
C_{orig} = The unspiked sample concentration in ug/L or mg/kg
Spike Quantity = The spiked concentration in ug/L or mg/kg

18.4.2 Calculate precision between two spikes using the following equation:

$$\text{Relative Percent Difference} = \left| \frac{C_{MSD} - C_{MS}}{\frac{C_{MSD} + C_{MS}}{2}} \right| \times 100$$

where:
C_{MSD} = The MSD concentration as read from the instrument printout in ug/L or mg/kg
C_{MS} = The MS concentration as read from the instrument printout in ug/L or mg/kg

18.4.3 Matrix spike recovery must be within 70-130% of the expected recovery. However, statistical limits will be used when available but must not exceed 70 – 130%. MSD relative percent difference must be ≤20%.

18.4.4 If MS/MSD criteria fail, reanalyze once. If the second attempt fails, initiate corrective action:

18.4.4.1 Verify the instrument is operating correctly.

18.4.4.2 Verify all other quality control is acceptable.

18.4.4.3 Re-digest the sample and MS/MSD and/or narrate in accordance with TriMatrix SOP GR-10-106.

18.5 Calculate CRL recovery as follows:

$$\text{Percent Recovery} = \frac{C_{Spike}}{\text{Spike Quantity}} \times 100$$

where:

C_{spike} = The analyzed CRL concentration in ug/L
Spike Quantity = The CRL concentration (0.2 ug/L)

18.6 The Method of Standard Additions (MSA)

18.6.1 Use the method of standard additions for all TCLP extracts having 50% or less matrix spike recovery.

18.6.2 Use the method of standard additions on samples from at least one waste stream of an F-waste delisting petition project. If Section 18.6.1 is not met however, apply the method of standard additions to all samples in the delisting.

18.6.3 Standard addition involves adding known mercury concentrations to three sample aliquots. These aliquots are digested and analyzed. The unspiked sample concentration and three spiked aliquots are used to construct a linear regression curve. The curve is extrapolated to find the point where it crosses the x axis. The absolute value of this point is the sample mercury concentration.

18.6.4 Prepare three matrix spikes at 50%, 100%, and 150% of the mercury concentration found in the sample. Final volumes must be the same for each.

18.6.5 Digest and analyze to obtain mercury results for each (in ug/L). Using the MSA regression spreadsheet located on the library drive of the facility network, plot absorbance vs. concentration in ug/L. Construct a linear curve without forcing through the origin. The extrapolated x-intercept point is the sample mercury concentration. An example of an MSA plot is shown in Attachment 23.4. For results to be valid, the following limitations must be taken into consideration:

18.6.5.1 The regression curve must be linear, with a correlation coefficient of ≥0.995 and with a slope less than or equal to 20% of the external calibration curve slope.

18.6.5.2 The chemical form of the mercury added must respond the same way as the mercury in the sample.

18.6.5.3 The interference effect must be constant over the working range of concern.

18.6.5.4 The method of standard additions will not correct for additive interference.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate an ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. A Continuing Demonstration of Capability (CDC) study is also required annually.

19.1.1 Initial Demonstration of Capability (IDC) Study

19.1.1.1 Prepare four second-source blank spikes at a concentration of 2.5 ug/L. Digest, filter and analyze following every step in the procedure.

19.1.1.2 Input the four results to the IDC spreadsheet located on the laboratory intranet library.

19.1.1.3 Average percent recovery must fall within 90 – 110% recovery. Standard deviation of the average must be ≤20% RSD.

19.1.1.4 If either criterion is not met, locate and correct the source of the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure or techniques used. If this occurs, locate and correct the source of the problem, document changes to the procedure and/or techniques used then repeat the study successfully.

19.1.1.5 Samples may not be analyzed by any analyst until a demonstration of capability study has been successfully completed.

19.1.1.6 Give a copy of successful IDC spreadsheets and raw data to the quality assurance department for training documentation.

19.1.2 Continuing Demonstration of Capability (CDC) Study

19.1.2.1 The demonstration of capability study must be repeated annually.

19.1.2.2 The CDC may be accomplished in one of the following ways:

19.1.2.2.1 By repeating the IDC study

19.1.2.2.2 By using the last four results from an MDL study performed exclusively by the analyst during the course of routine sample analysis. Only the last four results may be used.

19.1.2.2.3 By using four consecutive blank spike results run during the course of routine sample analysis, exclusively by the analyst.

19.1.2.2.4 By successfully and exclusively analyzing a single-blind performance testing sample during the course of routine sample analysis.

19.2 A method detection limit (MDL) study must be completed annually in accordance with TriMatrix SOP GR-10-125 for each digestion procedure.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material Safety Data Sheets are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"

22.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994

22.3 *Test Method for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision I, September 1994, Method 7471A, *"Mercury in Solid and Semi Solid Waste* (Manual Cold-Vapor Technique)"

23.0 ATTACHMENTS

23.1 Instrument Maintenance Logbook Example

23.2 Sample Identification and Weighing Sheet Example – Aqueous

23.3 MSA Curve Example

23.4 Internal Chain-of-Custody Example

23.5 Sample Identification and Weighing Sheet Example – Solid

Attachment 23.1
Instrument Maintenance Logbook Example

TriMatrix Laboratories, Inc.

PSA Maintenance Logbook

Date	Analyst	Emission/Reference Readings		Changed Pump Tubes	Changed Lamp	Changed Probe	Replaced Sample Valve	Comments
5/06/03	DW							Replaced dryer tube
05/06/03	MSS	196.4	108.8	✓				
05/08/03	DWS/MSS	200.1	110.4	✓				
05/08/03	MSS	185.9	114.1	✓				
05/9/03	MSS	196.1	112.1	✓				
05/13/03	MSS	195.1	114.1	✓				
05/14/03	MSS	180.4	112.2	✓				
05/15/03	MSS	193.1	113.1	✓				
05/16/03	MSS	193.9	111.3	✓				Replaced GLS line & Dryer Tube
05/20/03	MSS	200.1	113.2	✓				
05/21/03	MSS	200.0	112.7	✓				
05/24/03	MSS	180.9	107.9	✓				
05/28/03	MSS	199.5	107.1	✓				
05/29/03	MSS	184.1	110.9	✓				replaced GLS line & Dryer Tube
06/05/03	MSS	180.3	107.9	✓				
06/06/03	MSS	[illegible]	114.0	✓				
06/09/03	MSS	200.1	115.0	✓				
06/11/03	MSS	200.2	114.8	✓				
06/12/03	MSS	200.5	115.1	✓				

file: MaintAvalon.xls page: 3 of 25 revision: 1.0

Attachment 23.2
Sample Identification and Weighing Sheet Example – Aqueous

TriMatrix Laboratories, Inc.

Metals Laboratory Instrument Run Sequence Hg Aqueous

Instrument #: 216 — Data File:
Date: — Calibration BLK/ICB/CCB: 0.5% HNO3 LOT#1108080
Analyst: — Std/SCV prep date/analyst:
SnCl #: 8110207

Standards/SCV prepped daily
Autosampler Position:

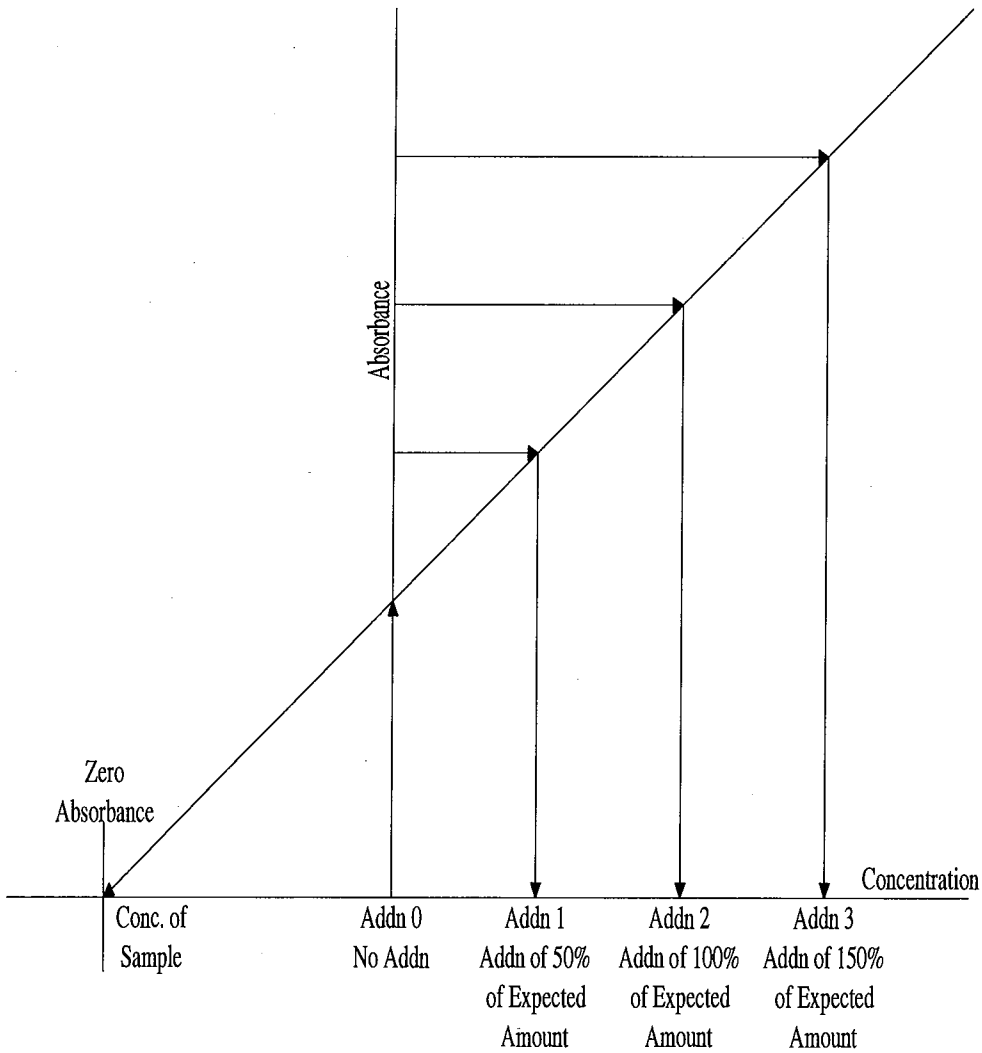
1. BLK/ICB/CCB/8050775
2. STD1/CRL/0.2 ug/L 8050776
3. STD2/0.5 ug/L 8050777
4. STD3/1.0 ug/L 8050778
5. STD4/CCV/2.0 ug/L 8050780
6. STD5/5.0 ug/L 8050781
7. SCV/3.0 ug/L 8100311

Position #	ID	Dilution			Position #	ID	Dilution		
		Initial Volume (mL)	Final Volume (mL)	Dilution Factor			Initial Volume (mL)	Final Volume (mL)	Dilution Factor
11					45				
12					46				
13					47				
14					48				
15					49				
16					50				
17					51				
18					52				
19					53				
20					54				
21					55				
22					56				
23					57				
24					58				
25					59				
26					60				
27					61				
28					62				
29					63				
30					64				
31					65				
32					66				
33					67				
34					68				
35					69				
36					70				
37					71				
38					72				
39					73				
40					74				
41					75				
42					76				
43					77				
44					78				

Hg_AQ.xls — revision 1.0

Approved By: 1-15-09 — **QA Officer** — **Approved By:** 1-19-09 — **Area Supervisor**

Attachment 23.3
MSA Curve Example



Attachment 23.4
Internal Chain-of-Custody Example

14-MAY-2003

TRIMATRIX LABORATORIES, INC.
CHAIN OF CUSTODY REPORT FOR AN ENTIRE SUBMITTAL

PAGE #6

CLIENT: MDEQ-RRD
PROJECT: Former Osceola Refinery West Branch-Sag. Bay Dist.
SUBMITTAL: May 7 & 8, 2003 Soils
PROJECT: 35923-7
Parameter: MERCURY, TOTAL
Method: CV/HG/7471/SOL
Ref Cit: USEPA-7471A
Matrix: SOIL

SAMPLE #	REMOVED BY: (SIGNATURE)	DATE & TIME REMOVED	RELINQUISHED BY: DATE & TIME	RECEIVED BY: DATE & TIME	DATE & TIME RETURNED
331322	M.A. Scott			05-15-03 1:30 pm	05-15-03 3:30 pm
331323	MSS				
331324	MSS				
331325	MSS				
331326	MSS				
331327	MSS				
331328	MSS				
331329	MSS				
331330	MSS				
331331	MSS				
331332	MSS				
331333	MSS				
331334	MSS				
331335	MSS				
331336	MSS				
331337	MSS			05-13-03 1:30 pm	05-15-03 3:30 pm

Approved By: [signature] 1-15-09
QA Officer

Approved By: [signature] 1-29-09
Area Supervisor

gr01123 5.6.doc

Attachment 23.5
Sample Identification and Weighing Sheet Example – Solid

TriMatrix Laboratories, Inc.

Metals Laboratory Instrument Run Sequence Hg Solids

Instrument #: 216 **Data File:**

Date: **Calibration BLK/ICB/CCB:** 0.5% HNO3 LOT#1108080

Analyst: **Std/SCV prep date/analyst:**

SnCl#: 8110207

Standards/SCV prepped daily
Autosampler Position:

1. BLK/ICB/CCB/8050782
2. STD1/CRL/0.2 ug/L 8050784
3. STD2/0.5 ug/L 8050786
4. STD3/1.0 ug/L 8050788
6. STD4/CCV/2.0 ug/L 8050789
6. STD5/5.0 ug/L 8050790
7. SCV/3.0 ug/L 8100310

Position #	ID	Dilution			Position #	ID	Dilution		
		Initial Volume (mL)	Final Volume (mL)	Dilution Factor			Initial Volume (mL)	Final Volume (mL)	Dilution Factor
11					45				
12					46				
13					47				
14					48				
15					49				
16					50				
17					51				
18					52				
19					53				
20					54				
21					55				
22					56				
23					57				
24					58				
25					59				
26					60				
27					61				
28					62				
29					63				
30					64				
31					65				
32					66				
33					67				
34					68				
35					69				
36					70				
37					71				
38					72				
39					73				
40					74				
41					75				
42					76				
43					77				
44					78				

Hg_solid.xls revision: 1.0

Approved By: 1-15-09 **QA Officer** **Approved By:** 1-19-09 **Area Supervisor**

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Inductively Coupled Plasma-Mass Spectrometry
Perkin Elmer Elan 6000/6100

SW-846 Method 6020A

UNCONTROLLED COPY

APPROVALS:

Area Supervisor: Denise S. Coffey Date: 2-22-10
Denise S. Coffey

QA Officer: Date: 2-22-10
Tom C. Boocher

Operations Manager: Date: 2/23/10
Jeff P. Glaser

Procedure Number: GR-01-129
Revision Number: 3.9

Date Initiated: 12/4/97
Effective Date: 3/10/10

Date Revised: 2/22/10
Pages Revised: All

By: Denise S. Coffey

Total Number of Pages: 34

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) is applicable to a large range of metallic elements in numerous matrices, including but not limited to soil, non-potable water, TCLP extracts, oil, solvents, sludge and air. Most matrices require solubilization or digestion prior to analysis.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*, 3rd Edition, Draft Update IVA, May 1998, Method 6020A, "Inductively Coupled Plasma – Mass Spectrometry", Revision 1, January 1998

3.0 SUMMARY OF PROCEDURE

3.1 Prior to analysis, samples are solubilized or digested using appropriate sample preparation. Refer to TriMatrix standard operating procedures for metals sample preparation and digestion.

3.2 This procedure describes the multi-elemental determination of analytes by ICP-MS. The procedure measures ions produced by a radio frequency inductively coupled plasma. Sample material in solution is introduced by pneumatic nebulization into radio-frequency plasma where energy transfer processes cause desolvation, atomization and ionization. Ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of mass-to-charge ratio by a quadrupole. Ion detection is by an electron multiplier or Faraday detector and the information is processed through data handling software.

3.3 Interferences must be assessed and valid corrections applied, or the data flagged to indicate problems. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents or from the sample matrix. Instrumental drift, suppressions or enhancements of instrument response caused by the sample matrix must be corrected by the use of internal standards.

4.0 PARAMETER OR COMPOUND LIST

4.1 This procedure may be used for analysis of the following metals.

Aluminum	Barium	Cadmium	Copper	Molybdenum	Silver	Vanadium	Strontium
Antimony	Beryllium	Chromium	Lead	Nickel	Thallium	Zinc	Gold
Arsenic	Boron	Cobalt	Manganese	Selenium	Tin	Uranium	Zirconium

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

UNCONTROLLED COPY

5.2 ELAN 6000 *Software Guide*; Perkin Elmer Corporation, Manual 0993-8965, Release E, June 1995

5.3 ELAN 6000 *Hardware Guide*; Perkin Elmer Corporation, Manual 0993-8969, Release D, September 1995

5.4 TriMatrix SOP GR-01-137, *Acid Digestion of Sediments, Sludges, Soils and Organic Waste using Block Digestion*, latest revision

5.5 TriMatrix SOP GR-01-144, *Block Digestion of Antimony in Sediments, Sludges and Soils*, latest revision

5.6 TriMatrix SOP GR-01-147, *Acid Digestion of Total/Dissolved Water TCLP/SPLP Extract and Wastewater by Block Digestion for ICP Total Metals Analysis Acid Digestion of Total/Dissolved Water and TCLP/SPLP Extract By Block Digestion for ICP/MS Total Metals Analysis*, latest revision

5.8 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

5.9 TriMatrix SOP GR-10-123, *Element™ Data Transfer and Review*, latest revision

5.10 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). All elements determined by this procedure have at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use, only molybdenum^{98} (ruthenium) and selenium^{82} (krypton) have isobaric elemental interferences. If alternative isotopes having higher natural abundance are selected to achieve greater sensitivity, an isobaric interference may occur. A data software system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element, and subtracting the appropriate signal from the analyte isotope signal. It should be noted that such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for calculations. Relevant isotope ratios must be established prior to the application of any corrections. Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, analysis using another verified and documented isotope, or use of another method.

6.2 Abundance sensitivity is a property defining the degree to which mass peak wings contribute to adjacent masses. Abundance sensitivity is affected by ion energy and quadropole operating pressure. Wing overlap interferences may result when a small ion peak is measured adjacent to a large one. The potential for these interferences must be recognized and spectrometer resolution adjusted to minimize them.

6.3 Isobaric molecular and doubly charged ion interferences in ICP-MS are caused by more than one atom or charge, respectively. Most isobaric interferences that affect ICP-MS determinations have been identified in the literature. Examples include $ArCl^+$ ions on the ^{75}As signal and MoO+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using natural

UNCONTROLLED COPY

isotope abundances from the literature, the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<2%) counting statistics. Because the ^{35}Cl natural abundance of 75.77% is 3.13 times the ^{37}Cl abundance of 24.23%, the chloride corrections for arsenic can be calculated (approximately) as follows, where the $^{38}Ar^{37}Cl+$ contribution at m/z is negligible 0.06% of the $^{40}Ar^{35}Cl+$ signal:

Corrected arsenic signal (using natural isotope abundances for coefficient approximations) =
[(m/z 75 signal)-((3.13)(m/z 77 signal))+((2.73)(m/z 82 signal))

Where the final term "((2.73)(m/z 82 signal))" adjusts for any selenium contribution at m/z 77

6.3.1 Cadmium values will be biased low by this type of equation when $^{97}ZrO^+$ ions contribute at m/z 108, but m/z 111 for cadmium is even subject to direct ($^{94}ZrO^+$) additive interferences when Zr is present.

6.3.2 The coefficients in the cadmium equation are ONLY illustrative for the arsenic equation above. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for the standard solution at a concentration providing suitable (<1%) counting precision.

6.3.3 The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable. For example, oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by use of an appropriate oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas has been shown to greatly reduce molecular interferences. These techniques can be used provided reporting limits, accuracy and precision requirements for analysis can be met.

6.3.4 If analysis requirements are not met, ionic interference is corrected based on actual instrument response instead of literature equations. Instrument-derived equations may be updated as needed without change to the standard operating procedure.

6.3.5 Ionic interference corrections are printed with each quantitative analysis calibration report after the correlation coefficient report. Refer to Attachment 23.11 for an ionic interference correction report example.

6.4 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.

UNCONTROLLED COPY

6.5 Memory interferences – Result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones and from material buildup in the plasma torch and spray chamber. The site where these effects occur is element dependent and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interference must be recognized within an analytical run and suitable rinse times used to reduce them. Memory interferences must be monitored within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alert to the possibility of a memory effect and must examine analyte concentration in the previous sample to identify if this was high. If memory interference is suspected, the sample must be reanalyzed after a long rinse period.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all standard operating procedures for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Concentrated acids are used in the preparation of standards and samples for analysis by ICP-MS. Disposable gloves, laboratory coats and approved safety glasses must be worn at all times when handling concentrated acids. Disposable gloves must also be worn when handling digested samples. Refer to the MSDS for information on these or any other chemical utilized in this procedure.

7.3 Check the exhaust hood over the instrument to be sure it is operating correctly. If the ventilation system is not working properly, extinguish the plasma if lit and immediately contact the metals laboratory manager. Under no circumstances must the ICP-MS be used if the exhaust hood is not working.

7.4 The plasma emits UV radiation. Avoid looking directly at the plasma without some type of strong UV protection. The instrument uses a UV resistant material in the viewing port so the analyst may watch the plasma. Do not tamper with this plate. Do not operate the machine without this plate in place. Do not attempt to view the plasma directly or indirectly in any way. Failure to follow this policy may cause serious and immediate damage to the retina of the eye and will result in disciplinary action.

7.5 The ICP-MS emits a strong RF field. To minimize exposure to this field, Perkin Elmer has included several safety interlocks to prevent direct exposure to harmful radiation. Never override any interlock on the ICP-MS. When working on the instrument, replace all RF shielding using all supplied screws. If any safety device has been tampered with, contact the metals lab manager.

7.6 The ICP uses argon to sustain plasma and to nebulize sample. Argon can cause suffocation through oxygen replacement. It is therefore imperative that all sources of argon be turned off with a valve when not in use. Since argon is colorless and odorless, if a feeling of lightheadedness develops, evacuate the metals laboratory at once and notify the area supervisor and/or the health and safety officer. Refer to the MSDS for information on this or any chemicals utilized.

7.7 All elements used in this procedure are toxic. Refer to the MSDS for information on these or any other chemicals utilized in this procedure.

UNCONTROLLED COPY

7.8 No food or drink is allowed in the metals laboratory.

7.9 Wash hands before starting work. Chemicals may be present on the skin, which may interfere with metals analysis. Wash hands before leaving the metals laboratory. Chemicals and acids may be on the skin that could eventually be ingested or passed on to a second party through casual contact.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Aqueous samples should have been acidified at the time of collection to a pH of <2. If the pH is not <2 when received by the laboratory, the sample pH must be lowered to <2 during the sample receipt process. A minimum of 24 hours must elapse after adjustment prior to sample digestion.

8.2 Solid samples require no preservation for metals. However, if the same sample container is scheduled for other analyses where refrigeration of the sample is needed, store at 4 ±2° C.

8.3 Samples may be collected in glass or polyethylene. If Silicon or Boron is to be analyzed, polyethylene containers must be used. Acidified sample must never come into contact with metal, to prevent contaminants leaching into the sample.

8.4 For dissolved metals analysis, the sample should have been filtered on site and acidified to pH <2. If pH is not <2, the sample pH must be lowered to <2 at sample receipt and a minimum of 24 hours must elapse after the adjustment, before analyzing the sample.

8.5 Holding times for all metals for ICP-MS is 180 days. In the case of TCLP or similar extracts, the holding time starts at the time leachate is generated.

8.6 The minimum sample volume for this procedure is 50 mL and 150 mL if samples require matrix QC. Samples must be solubilized prior to analysis. A smaller minimum volume is required if a sample will need diluted due to high concentration. Small minimum volumes sacrifice the reporting limit.

8.7 Digested (solubilized) samples and undigested acidified aqueous samples need not be refrigerated. All samples must be at room temperature prior to digestion. All digestates must be at room temperature prior to analysis.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Perkin Elmer, ELAN 6000/6100 inductively coupled plasma mass spectrometers (ICP-MS):

9.1.1 Both instruments are capable of scanning the mass range 6-240 amu with a minimum resolution of 1 amu peak width at 10% peak height. Both instruments are fitted with an extended dynamic range detection system. The instruments also include an ELAN data system capable of making corrections for isobaric interferences and application of the internal standards technique.

UNCONTROLLED COPY

NOTE: Electron multiplier detectors are used and precautions must be taken to prevent exposure to high ion flux. Otherwise changes in instrument response or damage to the multiplier may result.

9.1.2 Radio-frequency generators are used, compliant with FCC regulations.

9.1.3 A variable-speed peristaltic pump delivers solution to the nebulizer.

9.1.4 A mass-flow controller on the nebulizer gas supply is used.

9.2 Analytical balance with the capability of measuring to 0.1 mg for use in weighing solids, for preparing standards and for determining dissolved solids in digestates or extracts.

9.3 A Recirculator/chiller (Neslab CFT750). Temperature must be maintained at 18° C and pressure must be 35-60 psi. The recirculator is attached directly to the mass spectrometer to maintain isothermal conditions.

9.4 Centrifuge tubes (15 mL, clear) or equivalent. The tube must stand upright within the autosampler rack without tipping or leaning.

9.5 Eppendorf micropipettes capable of delivering 5-1000 uL

9.6 Flasks, Class A volumetric, various sizes

9.7 Pipettes, Class A, glass, various sizes

9.8 Balance, top-loading, capable of measurement to two decimal places.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 All maintenance activities must be recorded in the instrument maintenance logbook. Refer to Attachment 23.1 for an example instrument maintenance logbook.

10.2 At the beginning of each shift (every 8 hours) the manifold pump tubing must be replaced.

10.3 There are two filters on the ELAN 6000/6100 that must be replaced periodically. The two filters are located on the back of the instrument.

10.4 Inspect the waste container on the floor by the instrument every shift. If the jug is full, properly dispose of the liquid.

10.5 Visually inspect the torch before lighting the instrument. Do not touch or move the torch. The inspection must be visual only.

10.5.1 Open the vacuum chamber.

UNCONTROLLED COPY

10.5.2 Observe the torch. If the torch is broken or cracked, replace it. Refer to Section 10.6 for discussion on how to assemble and disassemble the torch.

10.5.3 Listen for obvious leaks in the argon lines going to the torch and nebulizer. If leaks are heard, investigate them and tighten the fittings to stop the leak. If a leak cannot be found or cannot be stopped, contact the metals laboratory manager.

10.6 Once every four to eight weeks disassemble the entire torch assembly and inspect all parts for wear. Replace all worn or damaged parts. Record all changes in the instrument maintenance logbook.

10.6.1 Turn off the plasma if it is on by selecting instrument, front panel, and then stop on the plasma box. The pump will stop when the plasma is extinguished.

10.6.2 Turn off the RF Generator by turning off the circuit breaker labeled RF Generator (CB1) on the left side of the instrument.

10.6.3 Loosen the retaining ring and remove the spray chamber assembly.

10.6.4 Open the top left cover of the ELAN. Use a flat blade screwdriver on the release mechanism to release the vacuum chamber interlock lever. Grasp the lever and pull in counter clockwise direction to slide the vacuum chamber and interface away from the torch box.

10.6.5 Remove the gas line connections from the torch by loosening the Swagelock fittings. Slide the fittings and connection tubing up and off the torch.

10.6.6 To remove the torch assembly, rotate the torch mount 1/8 turn counter clockwise and remove the entire assembly through the right side of the torch box.

10.6.7 With one hand grasping the torch and the other holding onto the Lexan adapter, twist and pull back on the adapter to separate the adapter from the torch.

10.6.8 Inspect all O-rings and replace any that are cracked or damaged.

10.6.9 Remove the alumina injector. If it is dirty, wash with soap and water. Dry, then put in the muffle furnace at 700° C for 20 minutes.

10.6.10 If the torch is dirty, immerse in an ultrasonic cleaner containing 1% nitric acid. Rinse with metals laboratory reagent water. Replace any torch that cannot be cleaned or is damaged.

10.6.11 After having been cleaned or replaced, reposition and align the ICP torch as follows:

10.6.11.1 Slide the injector/support adapter to the back of the torch.

10.6.11.2 Place the torch through the hole in the right side of the torch box. Rotate the torch assembly 1/8 turn clockwise until the bayonet mount is fully seated.

UNCONTROLLED COPY

10.6.11.3 Reattach the gas lines to the ICP torch. Slide both fittings and connecting tubing onto the torch and tighten the Swagelock fittings several turns.

10.6.11.4 To check the torch position and measure for proper alignment, follow Sections 10.6.11.5 through 10.6.11.8.

10.6.11.5 Place the torch alignment tool on the torch so the alignment tool flange touches the first turn of the load coil. Loosen the torch locking collar approximately 1 cm and slide the torch until it lines up with the outer edge of the alignment tool.

10.6.11.6 Slide the tool out approximately 1/2 inch and using the lever, move the vacuum chamber interlock into the lock position.

10.6.11.7 Move the vacuum chamber to the closed position to locate the alignment tool within the load coil.

10.6.11.8 Move the chamber to the open position and adjust the vacuum chamber position so the top end of the torch is even with the 5.5 mm cutout depth of the alignment tool.

10.6.11.9 If the torch is not properly lined up, loosen the two locking screws of the vacuum chamber interlock. Adjust the vacuum chamber forward or backward until the proper position is achieved.

10.6.11.10 Slide the vacuum chamber and interface toward the torch box. Secure the vacuum chamber interlock and close the top cover.

10.6.11.11 Turn on the RF Generator by turning on the circuit breaker labeled RF Generator (CB1) on the side of the instrument.

10.6.12 Inspect the drain line at the base of the spray chamber. There must be no leaks or cracks in the tubing. If a problem exists, replace the O-ring in the drain cap.

10.6.13 Inspect the nebulizer for wear.

10.6.13.1 If present, remove salt deposits with water.

10.6.13.2 If a cross-flow nebulizer is used, check the tips for wear. Replace the tips if excessive wear is noted.

10.7 Inspect the print quality of the printer. If the print is hard to read or light in appearance, replace the printer cartridge with the appropriate cartridge.

11.0 CHEMICALS AND REAGENTS

11.1 Acids used in standards preparation and for sample processing must be trace metal grade or better. Redistilled acids may be used if demonstrated to be free of contamination.

UNCONTROLLED COPY

11.1.1 Concentrated Nitric acid, trace metal grade (Fisher catalog A509-212 or equivalent)

11.1.2 Concentrated Hydrochloric acid, trace metal grade(Fisher catalog A508 or equivalent)

11.2 Laboratory reagent water from the MilliQ water system.

11.3 Argon gas supply: Welding grade or better. This is plumbed from a liquid argon tank located outside of the building. Argon is used as the main plasma gas, the auxiliary plasma gas, the nebulizer carrier gas and the RF coil coolant.

12.0 STANDARDS PREPARATION

12.1 All standards must be matrix matched as closely as possible to samples and/or sample digestates. All stock standards expire one year after receipt or on the vendor's expiration date, whichever is earlier.

12.2 Two calibration stock solutions identified as QCA and QCB are purchased at certified concentrations of 100 mg/L containing the following elements.

HP2079-1-1L Solution A (QCA)	Aluminum	Beryllium	Chromium	Lead	Selenium	Thallium
	Arsenic	Boron	Cobalt	Manganese	Silver	Vanadium
	Barium	Cadmium	Copper	Nickel	Strontium	Zinc

HP2079-1-1L Solution B (QCB)	Antimony
	Molybdenum
	Tin

12.3 Second-source stock solutions used as the secondary calibration verification (SCV) are purchased as single-element certified solutions at concentrations of 1000 or 10,000 mg/L from an alternate source/vendor.

12.4 The exception to the above is that uranium, gold and zirconium stock calibration and second-source standards are both purchased as single-element certified solutions.

12.5 All working standards and working standard dilutions for the ICP-MS prepared from stock standard solutions expire 24 hours from the time of preparation.

12.6 Prepare all standards using metals-free laboratory reagent water acidified to 1% with trace-metals grade nitric acid (HNO_3). The acidified water is prepared by adding 10 mL concentrated HNO_3 to 1000 mL laboratory reagent water.

12.7 Prepare uranium, gold and/or zirconium 100 ug/L standards as follows:

12.7.1 Label one bottle "100 mg/L U, Au and/or Zr stock std" then tare on the analytical balance.

12.7.2 Transfer 50 g of 1% nitric acid to the bottle.

UNCONTROLLED COPY

12.7.3 Pipette 1.0 mL of 10000 mg/L U, Au and/or Zr stock standard into the bottle then add 1% nitric acid until the final mass is 100 g.

12.8 Prepare intermediate and working standards of QCA and/or QCB with U, Au and/or Zr using 1% nitric acid. Rinse nine 125 mL polyethylene bottles with acidified water.

12.8.1 Label one bottle 100 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 100 µL of each 100 mg/L standard (labeled QCA, QCB and U, Au and/or Zr) into the bottle. Add 1% nitric acid until the final mass is 100 g.

12.8.2 Label one bottle 50 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 50 µL of each 100 mg/L standard (labeled QCA, QCB and U, Au and/or Zr) into the bottle. Add 1% nitric acid until the final mass is 100 g.

12.8.3 Label one bottle 20 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 20 µL of each 100 mg/L standard (labeled QCA, QCB and U, Au and/or Zr) into the bottle. Add 1% nitric acid until the final mass is 100 g.

12.8.4 Label one bottle 10 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 10 µL of each 100 mg/L standard (labeled QCA, QCB and U, Au and/or Zr) into the bottle. Add 1% nitric acid until the final mass is 100 g.

12.8.5 Label one bottle 1.0 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 1000 µL of the 100 ug/L standard into the bottle. Add 1% nitric acid until the final mass is 100 g.

12.8.6 Label one bottle 0.2 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 200 µL of the 100 ug/L standard into the bottle. Add 1% nitric acid until the final mass is 100 g.

12.8.7 Label one bottle 0.04 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 40 µL of the 100 ug/L standard into the bottle. Add 1% nitric acid until the final mass is 100 g.

12.8.8 Label one bottle a blank. Tare the bottle on the analytical balance. Pour 100 g of 1% nitric acid into the bottle.

12.8.9 Record all standard preparations in element (refer to Attachment 23.2).

12.9 Prepare Interference Check Standard A (IFA) as follows:

12.9.1 The IFAs are prepared to contain known concentrations of interfering elements that demonstrate the magnitude of interferences and provide an adequate test of any correction of false positives. Chloride in the IFA provides a means to evaluate software corrections for chloride-related interferences such as $^{35}Cl^{16}O^{+}$ on $^{51}V^{+}$ and $^{40}Ar^{35}Cl^{+}$ on $^{75}As^{+}$. Iron is used to demonstrate adequate resolution of the spectrometer for manganese determinations. Molybdenum serves to indicate oxide effects on cadmium isotopes. Other components are present to evaluate the

UNCONTROLLED COPY

ability of the measurement system to correct for various molecular-ion isobaric interferences. The IFA is used to verify that interference levels are corrected by the data system, within quality control limits.

12.9.2 Stock IFA solution is currently purchased from Inorganic Ventures with the following concentrations:

12.9.2.1 6020ICS-OA contains 10,000 mg/L Cl^-, 2000 mg/L C, 1000 mg/L Al, Ca, Fe, Mg, P, K, Na, S, 20 mg/L Mo and Ti as interferents.

12.9.3 Working IFA and IFB solutions must be prepared fresh daily.

12.9.3.1 IFA is prepared by pipetting 100 µL of 6020ICS-OA to 9.9 mL of 1% nitric acid.

12.9.3.2 IFB is prepared by pipetting 100 µL of 6020ICS-OA and 20 µL of each SCV stock prepared in section 12.10 to 9.9 mL of 1% nitric acid.

12.10 Prepare an SCV Stock Standard Solution as follows:

12.10.1 Rinse three 125 mL polyethylene bottle with 1% nitric acid and tare on the analytical balance.

12.10.2 Transfer approximately 50 g of 1% nitric acid into the bottles.

12.10.3 Pipette 100 µL of each of the following 10000 mg/L second source stock standards into first bottle:

Aluminum	Barium	Cadmium	Copper	Nickel	Thallium	Zinc
Arsenic	Beryllium	Chromium	Lead	Selenium	Silver	Uranium
Strontium	Boron	Cobalt	Manganese	Vanadium	Gold	Zirconium

12.10.4 Pipette 100 µL of 10000 mg/L second source stock standards Antimony and Molybdenum into second bottle.

12.10.5 Pipette 100 µL of 10000 mg/L second source stock standard Tin into the third bottle.

12.10.6 Dilute all three bottles to 100 g with 1% nitric acid.

12.10.7 Concentration for each element is 10 mg/L.

12.11 Prepare the SCV working solution as follows:

12.11.1 Rinse a 125 mL polyethylene bottle with 1% nitric acid.

12.11.2 Tare the bottle on an analytical balance.

12.11.3 Transfer 50 g of 1% nitric acid into the bottle.

UNCONTROLLED COPY

12.11.4 Pipette 400 µL of each SCV Stock Standard solution into the bottle then add 1% nitric acid until the final mass is 100 g.

12.11.5 Refer to Attachment 23.6 for concentrations.

12.12 Prepare Internal Standard Solution as follows:

12.12.1 For the ELAN 6000, pipette 2000 µL Lithium, 400 µL Germanium, 40 µL Indium, 40 µL Terbium, 60 µL Bismuth, 60 µL Rhodium and 40 µL Holmium into 1000 mL of 1% nitric acid.

12.12.2 For the ELAN 6100, pipette 2000 µL Lithium, 300 µL Germanium, 30 µL Indium, 20 µL Terbium, 40 µL Bismuth, 30 µL Rhodium and 20 µL Holmium into 1000 mL of 1% nitric acid.

12.12.3 Internal standard concentrations may be changed at any time, if changing the concentration generates a more appropriate intensity.

12.13 Prepare tuning intermediate solution as follows:

12.13.1 Rinse a 100 mL polyethylene bottle with 1% nitric acid and tare on the analytical balance.

12.13.2 Transfer approximately 50 g 1% nitric acid.

12.13.3 Pipette the following standards into the bottle:

12.13.3.1 100 µL of 10000 mg/L thallium

12.13.3.2 100 µL of 10000 mg/L lithium

12.13.3.3 100 µL of 10000 mg/L cobalt

12.13.3.4 100 µL of 10000 mg/L barium

12.13.3.5 100 µL of 10000 mg/L cerium

12.13.4 Add 1% nitric acid to a final mass of 100 g. Cap the bottle and shake.

12.13.5 Rinse a 1000 mL polyethylene bottle with 1% nitric acid and tare on the top-loading balance.

12.13.6 Transfer approximately 500 g 1% nitric acid.

12.13.7 Pipette the following standards into the bottle:

12.13.7.1 10 µL of 1000 mg/L indium

12.13.7.2 1000 µL of 6020A intermediate solution

UNCONTROLLED COPY

12.13.8 Add 1% nitric acid to a final mass of 1000 g. Cap the bottle and shake.

13.0 SAMPLE PREPARATION

13.1 Soil, sludge and waste samples must be digested in accordance with TriMatrix SOP GR-01-137 and GR-01-144.

13.2 Aqueous samples that need digested such as groundwater, TCLP and SPLP leachates must be digested in accordance with TriMatrix SOP GR-01-147.

13.3 Air samples are digested as specified by the method used in sample collection.

13.4 Any dissolved metals sample having an odor, precipitate or turbidity greater than 1 NTU after filtering must be digested prior to analysis.

14.0 CALIBRATION PROCEDURES

14.1 The ELAN 6000 and 6100 must be calibrated for every analytical run or every 24 hours, whichever is more frequent.

14.1.1 Initial calibration consists of an 8-point calibration curve for silver and cadmium. A 7-point curve is used for all other elements. An initial calibration curve consists of the standard series prepared in Section 12.4. Silver and cadmium curves include the 0.04 ug/L standard. This concentration is not included in the calibration for other elements. The 0 ug/L blank is included as a standard in all calibrations.

14.1.2 The average of at least three plasma responses for each concentration is used in generating calibration curves.

14.1.3 In all cases, calibration is performed using a multi-point linear regression. The correlation coefficient (r) must be greater than or equal to 0.995. If the coefficient is less than 0.995, recalibrate or postpone analysis (for only the out-of-control elements) until an acceptable calibration can be attained.

14.1.4 The highest concentration in the calibration range must not exceed 90% of the linear range (refer to Section 18.10).

14.2 After calibration, an Initial Calibration Verification is performed.

14.2.1 Analyze the Second-Source Calibration Verification (SCV). The SCV must be prepared from an independent (second source) material to a concentration near the mid-point of the calibration curve. The SCV control window is 90%-110% of the expected value. If the percent recovery of any target element does not fall within the control window, re-analyze one time. If the re-analysis does not pass recalibrate the instrument and restart analysis at Section 14.2 or postpone analysis (for only the out-of-control elements) until an acceptable calibration can be obtained.

UNCONTROLLED COPY

14.2.2 Analyze the Initial Calibration Blank (CCB). A CCB is quantitation of the 0 ug/L blank (prepared in Section 12.4.9), as a sample. The absolute value of the CCB reading must be less than 1/5th the minimum reporting limit required for analysis. If the CCB is not lower than 1/5th the reporting limit, re-analyze one time. If the re-analysis does not pass recalibrate the instrument and start again at Section 14.2 or postpone analysis (for only out-of-control elements) until an acceptable calibration can be generated. If the ICB remains out-of-control, see the metals laboratory supervisor.

14.2.3 Analyze the MRL-level Calibration Verification (CRL). A CRL is a reporting level standard with a concentration at the minimum reporting limit (MRL). The control window is 50%-150% of the expected value. If percent recovery falls outside the 50-150% window, a narrative must be attached to the analytical batch.

14.2.4 Analyze the IFA solution which is comprised of interfering elements only. The IFA demonstrates the magnitude of interferences and provides a test of any corrections made. Control limits for the IFA are ±3 times the reporting limit for non-spiked elements.

14.2.5 Analyze Interference Check Standard B (IFB). The IFB contains a known concentration of interfering elements that monitors analyte recovery and/or detection, and provides a test for any corrections that need done.

14.2.5.1 Control limits are 90-110% of the known concentration and ±3 times the reporting limit for non-spiked elements.

14.2.5.2 If the IFB is outside a control limit, stop the analysis and remake the solution then re-analyze successfully to continue with sample analysis.

14.2.5.3 If the IFB fails again, generate an acceptable calibration and repeat the analysis for all out-of-control elements. Alternatively, postpone analysis until an acceptable calibration can be generated.

14.2.6 Following the IFB, analyze a Continuing Calibration Verification (CCV) standard. A CCV is the same solution as the SCV. The control window is 90%-110% of the expected value. If the CCV is not within the control window, stop the analysis and re-analyze one time. If the CCV re-analysis is within the control window, continue the analysis. If the CCV remains out-of-control, recalibrate the instrument for the affected elements and restart the analysis at Section 14.2 or postpone analysis (for only out-of-control elements) until an acceptable calibration can be generated.

14.2.7 Follow the CCV with analysis of a Continuing Calibration Blank (CCB). A CCB is quantitation of the 0 ug/L blank prepared in Section 12.4.9, as a sample. The absolute value of the CCB reading must be less than 1/5th the lowest reporting limit required for analysis. If the CCB is not lower than 1/5th the reporting limit, stop the analysis and re-analyze one time. If the CCB re-analysis is less than 1/5th the reporting limit, continue sample analysis. If the CCB remains out-of-control, recalibrate the instrument for the affected elements and restart the analysis at Section

UNCONTROLLED COPY

14.2 or postpone analysis (for only out-of-control elements) until an acceptable calibration can be generated.

14.2.8 Analyze up to ten samples. If percent RSD for any result obtained from the replicate plasma response is >20, the sample run immediately prior to the result must be checked for high analyte concentration. If the analyte is high, check the triple-replicate result in question to verify that concentration is not decreasing. If concentration is decreasing, reanalyze the sample. If the concentration is not decreasing, report the result in question.

14.2.9 After each analysis of up to ten samples, analyze the CCV. If the CCV is not within the control window, stop the analysis and re-analyze one time. If CCV re-analysis is within the control window, continue sample analysis. If CCV re-analysis is out-of-control, recalibrate the instrument and start again at Section 14.2 or postpone analysis (for only out-of-control elements), until an acceptable calibration can be generated. All samples associated with an unacceptable CCV must be re-analyzed for all affected target elements.

14.2.10 After each CCV, analyze the CCB. If a CCB is not less than $1/5^{th}$ the reporting limit, stop the analysis and re-analyze one time. If CCB re-analysis is within the control window, continue sample analysis. If CCB re-analysis is out-of-control, recalibrate the instrument and start again at Section 14.2 or postpone analysis (for only out-of-control elements), until an acceptable calibration can be generated. All samples associated with an unacceptable CCB must be re-analyzed for all affected target elements.

14.2.11 Repeat Sections 14.2.8 through 14.2.10 until all samples have been run. The analytical run must be concluded with analysis of acceptable CCV and CCB results.

14.2.12 The IFA and IFB solutions must be re-verified if an analytical run exceeds 12 hours.

15.0 ANALYTICAL PROCEDURE

15.1 The following sections assume the user to be familiar with ELAN 6000/6100 operating software. If unfamiliar or unsure of any particular part of the operation, consult the instrument manual for additional instructions. This section provides adequate instruction for normal instrumental operation but is not intended to replace the instrument operating manual.

15.2 Preliminary treatment of all sample matrices is almost always necessary to solubilize analyte.

15.2.1 Digestions are referenced in Section 5.0.

15.2.2 A minimum of five internal standards must always be used.

15.3 Once the plasma has been on for at least 30 minutes, daily tuning of the instrument needs performed. However, if the sample and skimmer cones have been changed, a more rigorous tune needs performed as follows:

15.3.1 Realign the x and y cone axis.

UNCONTROLLED COPY

15.3.2 Optimize the nebulizer flow and lens voltage.

15.3.3 Tune the mass spec over the entire mass range.

15.3.4 Check instrument performance:

15.3.4.1 The instrument will take six readings to generate a percent relative standard deviation (RSD) value for lithium, cobalt, indium and thallium. For each element, RSD must be less than 5.0. If not, repeat the evaluation once. If any element is greater than or equal to 5.0, locate and correct the problem before proceeding.

15.3.4.2 Calculate Percent Ba^{2+}/Ba^{0} and CeO/Ce^{0}. If either criterion is greater than or equal to 3.0%, repeat the check once. If one or the other remains out of limits, locate and correct the problem then repeat the tune.

15.4 Once all tune optimization steps are complete, calibrate in accordance with Section 14.0 and run samples.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Collect data from the instrument and transfer to the laboratory information management system in accordance with TriMatrix SOP GR-10-123.

16.3 Whenever possible, calculate a result from both primary and secondary isotopes. Report only the primary isotope result. However, if there is greater than 25% difference between the two values, report the lowest value as the result. Monitor percent difference as follows:

$$\frac{\text{primary ion} - \text{secondary ion}}{\text{primary ion}} \times 100 = \text{percent difference}$$

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for correctly filling in, handing in and filing all associated paperwork. It is essential to perform these tasks conscientiously to provide clients with defensible data.

17.2 Report data to the laboratory information management system (Element™) in accordance with TriMatrix SOP GR-10-123.

17.2.1 Data must be recorded completely to ensure that results are reported correctly and data is associated with the right quality control batch. It is important that all required information be recorded correctly and completely.

17.2.2 Quality control information must be completed and recorded including spiked concentrations where applicable.

UNCONTROLLED COPY

17.2.3 If internal chain-of-custody (CoC) is required, it is important that CoC forms be filled in correctly and archived appropriately. Refer to Attachment 23.5 for an internal CoC form example.

17.2.4 All analysis hardcopy and benchsheets must be archived appropriately.

17.3 Laboratory Required Paperwork

17.3.1 All run and maintenance logbooks must be filled in completely and correctly. Corrections must be made with a lineout, not a write-over, and must be dated and initialed. Blank lines in logbooks must be Z'd out.

18.0 QUALITY ASSURANCE

18.1 All quality control data must be maintained and available for easy reference or inspection.

18.2 Instrument detection limit (IDL) studies must be performed quarterly.

18.2.1 Analyze seven sequential aliquots of metals reagent water. Repeat on two more non-consecutive days to obtain 21 results in 3 datasets.

18.2.2 Input these data to the IDL spreadsheet located on the laboratory intranet to calculate the IDL which is the calculated average standard deviation.

18.2.3 The spreadsheet compares the calculated IDL to the calculated MDL. The IDL must be less than the MDL. If not, locate the source of the problem and repeat the IDL/MDL studies successfully. Repeat only for elements that failed.

18.2.4 Consult the metals supervisor and/or the quality assurance department if repeated studies indicate the IDL is not less than the MDL.

18.3 Dilute and re-analyze samples that are more concentrated than the highest standard on the calibration curve.

18.4 Include a minimum of one Blank (BLK) per sample batch digested to determine if contamination or any memory effects are occurring. The BLK must be carried through all sample preparation steps. Results must be below the reporting limit.

18.5 Include a minimum of one Laboratory Control Sample (BS) per sample batch. The BS must be carried through all sample preparation steps. A BS is reagent water spiked with each element between the low and mid-level calibration range. Percent recovery is not to exceed 80%-120% recovery of the expected value. If a BS is outside the control window, it must be re-run one time. If the re-analysis is outside the control window all samples associated with the BS must be re-prepared and re-analyzed.

18.6 Analyze one matrix spike (MS) and one matrix spike duplicate (MSD) at a frequency of at least 1 in 20 (5%). MS/MSDs are sample aliquots to which is added a known analyte concentration between the low and mid-level calibration range. Calculate percent recovery for each element. Percent recovery is not to exceed

UNCONTROLLED COPY

75%-125% recovery with an RPD <20%. Matrix spiking monitors sample preparation efficiency relative to each matrix type. Relative percent difference (RPD) is calculated from the MS/MSD duplication. The RPD value monitors precision within sample preparation and analysis.

18.6.1 Pretreated samples must be spiked before digestion begins.

18.6.2 Samples not requiring pretreatment are spiked before loading on the autosampler tray.

18.6.2.1 Two "spiking solutions" are used to perform matrix spikes. These solutions are stable but due to high analyte concentrations are not compatible when mixed.

18.6.3 Calculate MS/MSD percent recovery as follows:

$$\%R = \frac{C_{Spike} - C_{Orig}}{\text{Spike Concentration}} \times 100$$

where:

%R	=	Spike recovery, in %
C_{Spike}	=	Concentration of the spiked sample
C_{Orig}	=	Concentration of the unspiked sample
Spike Concentration	=	Concentration of the element spiked into the sample

18.6.4 Calculate MS/MSD relative percent difference as follows,

$$\%RPD = \frac{C_{MSD} - C_{MS}}{(C_{MSD} + C_{MS})/2} \times 100$$

where:

%RPD	=	Relative percent difference between the MS and MSD
C_{MSD}	=	Concentration of the MSD as read from the instrument data printout
C_{MS}	=	Concentration of the MSD as read from the instrument data printout

18.6.5 A matrix spike must recover within 75-125% of the expected value. Precision between two matrix spikes must be ≤20%. Refer to TriMatrix SOP GR-10-106 for corrective actions.

18.7 The following tests must be performed with each sample batch to ensure that neither positive nor negative interferences are affecting element measurement or distorting reported value accuracies.

18.7.1 Post Spike (PS): The same sample from which the MS/MSD aliquots were prepared must also be spiked with a Post Spike. An analyte spike between the low and mid-range of the calibration curve is added to a portion of prepared digestate or digestate dilution. The control window is 80%-120% of the expected value. If MS/MSD recovery is acceptable and the PS is unacceptable, a serial dilution must be performed on the sample. If MS/MSD recovery is unacceptable and PS recovery is unacceptable then a matrix effect is confirmed and the sample qualified accordingly.

UNCONTROLLED COPY

18.7.2 Serial Dilution: A serial dilution must be performed on the same sample as the MS/MSD, if analyte concentration is ≥100 times the IDL. Analysis of a 1:5 dilution must agree within ±10% of the original determination. If not, a chemical or physical interference effect must be suspected and the sample qualified accordingly.

18.7.3 The method of standard additions involves adding known concentrations of a standard to three digestate aliquots. The three results are then used to construct a linear regression curve. The curve is extrapolated to find the point where it crosses the x-axis. The value of this point is the analyte concentration in the sample.

18.7.4 To perform the method of standard additions, prepare three spikes at 50%, 100% and 150% of the concentration found in the processed sample.

18.7.5 Analyze the spikes and the non-spiked processed sample. Using the regression program located on the laboratory intranet library, plot absorbance against concentration to construct a regression curve. The extrapolated x-intercept of the regression curve is the analyte concentration in the sample. Refer to Attachment 23.9 for a plot example. For results to be valid, the following limitations must be taken into consideration:

18.7.5.1 The curve constructed must be linear, with a correlation coefficient of ≥0.995.

18.7.5.2 The chemical form of the analyte added must respond the same way as the analyte in the sample.

18.7.5.3 The interference effect must be constant over the working range of concern.

18.7.5.4 The signal must be corrected for any additive interference.

18.7.5.5 The method of standard additions must be performed on any TCLP sample whose spike recovery is less than 50% and with a concentration less than or equal to the regulatory limit. The method of standard additions is also required when a TCLP extract concentration is within 20% of the regulatory limit. TCLP regulatory limits are as follows:

Element	Regulatory Limit (mg/L)	Element	Regulatory Limit (mg/L)
Arsenic	5.0 mg/L	Lead	5.0 mg/L
Barium	100 mg/L	Selenium	1.0 mg/L
Cadmium	1.0 mg/L	Silver	8.0 mg/L
Chromium	5.0 mg/L	Zinc	500 mg/L
Copper	100 mg/L		

UNCONTROLLED COPY

18.7.5.6 The method of standard additions does not detect coincidental spectral overlap. If overlap is suspected, use computer compensation, an alternative wavelength or comparison with an alternate method to determine and/or correct.

18.7.6 Duplicate Analysis (DUP): If specified for a project, a DUP is analyzed as a substitute for, or in addition to the MSD. A DUP is a second sample aliquot analyzed in the same way as the first. The acceptance limit for DUP RPD is ≤20%.

18.8 Verify instrument calibration by analyzing appropriate quality control verification standards as described in Section 14.0.

18.9 Internal standardization (IS) must be used in all analyses to correct for instrument drift and physical interferences. A minimum of five internal standards are used at all times. Recovery for each internal standard is 70-120% of the expected value.

18.9.1 If an internal standard has low recovery (<70%), do a 1:5 dilution of the sample or digestate and re-analyze. If internal standard recovery is below 70%, do another 1:5 dilution. Repeat until acceptable internal standard recovery is achieved.

18.9.2 If an internal standard recovery is high (>120%), the element may be present in the sample. Use an alternate internal standard.

18.9.3 All internal standards must be within 50 amu of the element of interest.

18.10 Linear range studies must be performed semi-annually or more frequently if specified by any project DQO, or when there is a significant change in instrument response.

18.10.1 To perform a linear range study, calibrate the instrument as for sample analysis.

18.10.2 Run successive standards at an increasingly higher concentration and calculate percent recovery for each element. Begin by running a 200 ug/L standard.

18.10.3 If percent recovery is acceptable (90-110%) for the 200 ug/L standard, run a higher concentration. Continue until percent recovery is not acceptable then run a standard at a lower concentration but higher than the last acceptable standard to zero in on the maximum value in the linear range.

18.10.4 The highest concentration run with acceptable percent recovery is the linear range.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

UNCONTROLLED COPY

19.1.1 Initial Demonstration of Capability

19.1.1.1 Spike four aliquots of metals reagent water with a second source standard so concentration is in the lower half of the calibration range.

19.1.1.2 Digest and analyze the four spikes as samples following every step in the procedures.

19.1.1.3 Input the four results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation. Average recovery must be within blank spike control limits. Relative standard deviation must be ≤20%.

19.1.1.4 If either criterion is not met, locate and correct the source of the problem and repeat the study successfully.

19.1.1.5 Repeated failure will confirm a general problem with the procedure and/or techniques used. If this occurs, locate and correct the source of the problem, revise the procedure and/or techniques uses then repeat the study successfully.

19.1.1.6 Samples may not be analyzed by any analyst or on any instrument until a successful demonstration of capability study has been completed.

19.1.2 A Continuing Demonstration of Capability (CDC) study must be performed annually by one of the following:

19.1.2.1 By repeating the IDC study.

19.1.2.2 By using four consecutively analyzed blank spikes (if a second source is used) obtained from routine sample analysis if run exclusively by the analyst.

19.1.2.3 By successfully analyzing a performance testing sample during the course of routine sample analysis if run exclusively by the analyst.

19.1.2.4 By using the last four of seven results from an MDL study if run exclusively by the analyst. ONLY the last four results may be used.

19.1.3 Submit copies of all successful demonstrations of capability spreadsheets to the quality assurance department for data and training documentation.

19.2 A Method Detection Limit Study must be performed annually in accordance with TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

UNCONTROLLED COPY

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located in the library on the laboratory intranet.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Draft Update IVA, May 1998, Method 6020A, "Inductively Coupled Plasma – Mass Spectrometry", Revision 1, January 1998

23.0 ATTACHMENTS

23.1 Instrument Maintenance Logbook Example

23.2 Standards Log Example

23.3 Preparation Batch Report Example

23.4 Data Review Report Example

23.5 Internal Chain-of-Custody Example

23.6 SCV Standard Solution

23.7 Element Conditions

23.8 Spiking Solution Final Concentration

23.9 MSA Curve Example

UNCONTROLLED COPY

23.10 Ionic Interference Correction Report Example

UNCONTROLLED COPY

Attachment 23.1
Instrument Maintenance Logbook Example

TriMatrix Laboratories, Inc.

ICP Instrument Maintenance Logbook

Date	Analyst	Changed Pump Tube	Torch		Nebulizer		Wavelength Calibration	Alumina Injector		Replaced RF Coil	Other/Comments
			Cleaned	Replaced	Cleaned	Changed Tips		Cleaned	Replaced		
2-6-01	KW										Changed cones
2/7/01	SAM	✓									-changed pump tubes -changed mist chamber, filter ✓
2/8/01	SAM	✓									
2/9/01	SAM	✓									changed cones.
2-12-01	KW	✓									
2/13/01	SAM	✓									
2/14/01	SAM	✓									Changed Cones
2-15-01	AW	7:25 AM									
2-19-01	AW	7:45 AM									
2-19-01	AW										Replaced gaskets
2-20-01	DSi	9:15am									
2/21/01	SAM	8:50am									
2/21/01	SAM	4:05pm									Changed Cones
2-21-01	KW	8:00pm									

UNCONTROLLED COPY

Attachment 23.2
Standards Log Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
A602735

Description:	QCA ICPMS	Expires:	Mar-31-07
Standard Type:	Analyte Spike	Prepared:	Mar-31-05
Solvent:	Solvent Lot #605319	Prepared By:	** Vendor **
Final Volume (mls):	1000	Department:	Expired
Vials:	1	Last Edit:	Jan-30-07 15:48 by DSC

Environmental Express part# HP2078-1-1L Solution A
Lot: 605319
Received 2/28/06

Analyte	CAS Number	Concentration	Units
Aluminum	7429-90-5	100	mg/L
Arsenic	7440-38-2	100	mg/L
Arsenic, OWEP-SAR	7440-38-2	100	mg/L
Arsenic, OWEP-TAR	7440-38-2	100	mg/L
Barium	7440-39-3	100	mg/L
Beryllium	7440-41-7	100	mg/L
Boron	7440-42-8	100	mg/L
Cadmium	7440-43-9	100	mg/L
Calcium	7440-70-2	1000	mg/L
Chromium	7440-47-3	100	mg/L
Cobalt	7440-48-4	100	mg/L
Copper	7440-50-8	100	mg/L
Iron	7439-89-6	100	mg/L
Lead	7439-92-1	100	mg/L
Lead, Total (Coarse)	7439-92-1	100	mg/L
Lead, Total (Fine)	7439-92-1	100	mg/L
Lithium	7439-93-2	100	mg/L
Magnesium	7439-95-4	1000	mg/L
Manganese	7439-96-5	100	mg/L
Nickel	7440-02-0	100	mg/L
Phosphorus	7723-14-0	100	mg/L
Potassium	7440-09-7	1000	mg/L
Selenium	7782-49-2	100	mg/L
Selenium, OWEP-SAR	7782-49-2	100	mg/L
Selenium, OWEP-TAR	7782-49-2	100	mg/L
Silver	7440-22-4	100	mg/L
Sodium	7440-23-5	1000	mg/L
Strontium	7440-24-6	100	mg/L
Thallium	7440-28-0	100	mg/L
Thallium, OWEP-SAR	7440-28-0	100	mg/L

Reviewed By: _____ Date: _____

Page 1 of 2

Analytical Standard Record
TriMatrix Laboratories, Inc.
A602735

Thallium, OWEP-TAR	7440-28-0	100	mg/L
Vanadium	7440-62-2	100	mg/L
Zinc	7440-66-6	100	mg/L

Reviewed By: _____ Date: _____

Page 2 of 2

UNCONTROLLED COPY

Attachment 23.3
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0609650 Page 1 of 2

Printed: 10/5/2007 3:04:45PM

Metals, Water, 3020A Digestion
(No Surrogate)
Batch Comments: 08/23/06 6:15am-2:30pm

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0608312	Se Total 6020	0608312	Pb Total 6020	0608312	Cr Total 6020
0608312	Cd Total 6020	0608312	As Total 6020	0608312	Ag Total 6020
0608366	Zn Total 6020	0608355	Se Total 6020	0608355	Pb Total 6020
0608355	Cu Total 6020	0608355	Cr Total 6020	0608355	Cd Total 6020
0608355	Ba Total 6020	0608355	As Total 6020	0608355	Ag Total 6020

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0609650-BLK1		Aug-23-06 06:15	LMA	25	125					BLANK	
0609650-BS1		Aug-23-06 06:15	LMA	25	125			A602735	25	LCS	
0609650-MS1		Aug-23-06 06:15	LMA	25	125		0608355-22	A602736	25	MATRIX SPIKE	
0609650-MSD1		Aug-23-06 06:15	LMA	25	125		0608355-22	A602736	25	MATRIX SPIKE DUP	
0608312-23	A	Aug-23-06 06:15	LMA	25	125					[redacted]	
0608312-23	A	Aug-23-06 06:15	LMA	25	125						
0608312-23	A	Aug-23-06 06:15	LMA	25	125						
0608312-23	A	Aug-23-06 06:15	LMA	25	125						
0608312-23	A	Aug-23-06 06:15	LMA	25	125						
0608312-23	A	Aug-23-06 06:15	LMA	25	125						
0608355-21	A	Aug-23-06 06:15	LMA	25	125						
0608355-21	A	Aug-23-06 06:15	LMA	25	125						
0608355-21	A	Aug-23-06 06:15	LMA	25	125						
0608355-21	A	Aug-23-06 06:15	LMA	25	125						
0608355-21	A	Aug-23-06 06:15	LMA	25	125						
0608355-21	A	Aug-23-06 06:15	LMA	25	125						
0608355-22	C	Aug-23-06 06:15		25	125						Added for SequenceQC in 0082023
0608355-22	C	Aug-23-06 06:15	LMA	25	125						
0608355-22	C	Aug-23-06 06:15	LMA	25	125						
0608355-22	C	Aug-23-06 06:15	LMA	25	125						

Comments:

Analyst Initials:

bdi_TriMatrix.rpt

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0609650 Page 2 of 2

Printed: 10/5/2007 3:04:45PM

Metals, Water, 3020A Digestion
(No Surrogate)
Batch Comments: 08/23/06 6:15am-2:30pm

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0608355-22	C	Aug-23-06 06:15		25	125					[redacted]	Added for SequenceQC in 0082023
0608355-22	C	Aug-23-06 06:15	LMA	25	125						
0608355-22	C	Aug-23-06 06:15	LMA	25	125						
0608355-22	C	Aug-23-06 06:15		25	125						Added for SequenceQC in 0082023
0608355-22	C	Aug-23-06 06:15	LMA	25	125						
0609650-PS1		Aug-28-06 13:15	DSC	2	10		0608355-22	A603106	50	POST SPIKE	[Spk] 25mL->125mL, 10mL->10mL, Spk
0609650-PS2		Aug-28-06 14:00	DSC	2	10		0608355-22	A603106	100	POST SPIKE	[Spk] 25mL->125mL, 10mL->10mL, Spk

Comments:

Analyst Initials:

bdi_TriMatrix.rpt

UNCONTROLLED COPY

Approved By: [signature] 2-22-10 **QA Officer**

Approved By: [signature] 2.22.10 **Area Supervisor**

Attachment 23.4
Data Review Report Example

TriMatrix Laboratories, Inc

Data Review Report -- Metals Lab
Sequence = 6090602 Page 1 of 4

SampleID	Analysis	IResult	Qualifier	IUnits	Dilu	FResult	FUnits	IMDL	IMRL	EM
6090602-CAL1	Ag Total 6020	0		ug/L	1	0.00	ug/L	0.024	0.04	
6090602-CAL1	Cu Total 6020	0		ug/L	1	0.00	ug/L	0.068	0.2	
6090602-CAL1	Pb Total 6020	0		ug/L	1	0.00	ug/L	0.065	0.2	
6090602-CAL1	Tl Total 6020	0		ug/L	1	0.00	ug/L	0.034	0.2	
6090602-CAL1	V Total 6020	0		ug/L	1	0.00	ug/L	0.17	0.2	
6090602-CAL2	Ag Total 6020	0.04		ug/L	1	0.0400	ug/L	0.024	0.04	
6090602-CAL3	Ag Total 6020	0.200592		ug/L	1	0.201	ug/L	0.024	0.04	
6090602-CAL3	Cu Total 6020	0.2		ug/L	1	0.200	ug/L	0.068	0.2	
6090602-CAL3	Pb Total 6020	0.2		ug/L	1	0.200	ug/L	0.065	0.2	
6090602-CAL3	Tl Total 6020	0.2		ug/L	1	0.200	ug/L	0.034	0.2	
6090602-CAL3	V Total 6020	0.2		ug/L	1	0.200	ug/L	0.17	0.2	
6090602-CAL4	Ag Total 6020	1.000281		ug/L	1	1.00	ug/L	0.024	0.04	
6090602-CAL4	Cu Total 6020	1.002342		ug/L	1	1.00	ug/L	0.068	0.2	
6090602-CAL4	Pb Total 6020	1.00034		ug/L	1	1.00	ug/L	0.065	0.2	
6090602-CAL4	Tl Total 6020	1.000458		ug/L	1	1.00	ug/L	0.034	0.2	
6090602-CAL4	V Total 6020	0.99968		ug/L	1	1.00	ug/L	0.17	0.2	
6090602-CAL5	Ag Total 6020	9.985816		ug/L	1	9.99	ug/L	0.024	0.04	
6090602-CAL5	Cu Total 6020	9.987423		ug/L	1	9.99	ug/L	0.068	0.2	
6090602-CAL5	Pb Total 6020	9.989133		ug/L	1	9.99	ug/L	0.065	0.2	
6090602-CAL5	Tl Total 6020	9.987984		ug/L	1	9.99	ug/L	0.034	0.2	
6090602-CAL5	V Total 6020	9.989488		ug/L	1	9.99	ug/L	0.17	0.2	
6090602-CAL6	Ag Total 6020	20.23713		ug/L	1	20.2	ug/L	0.024	0.04	
6090602-CAL6	Cu Total 6020	20.21078		ug/L	1	20.2	ug/L	0.068	0.2	
6090602-CAL6	Pb Total 6020	20.09089		ug/L	1	20.1	ug/L	0.065	0.2	
6090602-CAL6	Tl Total 6020	20.14292		ug/L	1	20.1	ug/L	0.034	0.2	
6090602-CAL6	V Total 6020	20.08358		ug/L	1	20.1	ug/L	0.17	0.2	
6090602-CAL7	Ag Total 6020	49.5229		ug/L	1	49.5	ug/L	0.024	0.04	
6090602-CAL7	Cu Total 6020	49.47732		ug/L	1	49.5	ug/L	0.068	0.2	
6090602-CAL7	Pb Total 6020	49.94873		ug/L	1	49.9	ug/L	0.065	0.2	
6090602-CAL7	Tl Total 6020	49.54875		ug/L	1	49.5	ug/L	0.034	0.2	
6090602-CAL7	V Total 6020	49.59068		ug/L	1	49.6	ug/L	0.17	0.2	
6090602-CAL8	Pb Total 6020	101.319		ug/L	1	101	ug/L	0.065	0.2	
6090602-ICV1	Ag Total 6020	42.35505		ug/L	1	42.4	ug/L	0.024	0.04	
6090602-ICV1	Cu Total 6020	42.45288		ug/L	1	42.5	ug/L	0.068	0.2	
6090602-ICV1	Pb Total 6020	41.42369		ug/L	1	41.4	ug/L	0.065	0.2	
6090602-ICV1	Tl Total 6020	43.27606		ug/L	1	43.3	ug/L	0.034	0.2	
6090602-ICV1	V Total 6020	40.95888		ug/L	1	41.0	ug/L	0.17	0.2	
6090602-SCV1	Ag Total 6020	42.35505		ug/L	1	42.4	ug/L	0.024	0.04	
6090602-SCV1	Cu Total 6020	42.45288		ug/L	1	42.5	ug/L	0.068	0.2	
6090602-SCV1	Pb Total 6020	41.42369		ug/L	1	41.4	ug/L	0.065	0.2	
6090602-SCV1	Tl Total 6020	43.27606		ug/L	1	43.3	ug/L	0.034	0.2	
6090602-SCV1	V Total 6020	40.95888		ug/L	1	41.0	ug/L	0.17	0.2	
6090602-ICB1	Ag Total 6020	0.009862		ug/L	1	0.00988	ug/L	0.024	0.04	
6090602-ICB1	Cu Total 6020	0.004933		ug/L	1	0.00493	ug/L	0.068	0.2	
6090602-ICB1	Pb Total 6020	0.003587		ug/L	1	0.00359	ug/L	0.065	0.2	
6090602-ICB1	Tl Total 6020	0.004911		ug/L	1	0.00491	ug/L	0.034	0.2	
6090602-ICB1	V Total 6020	0.016471		ug/L	1	0.0165	ug/L	0.17	0.2	
6090602-CRL1	Ag Total 6020	0.224835		ug/L	1	0.225	ug/L	0.024	0.04	
6090602-CRL2	Cu Total 6020	1.146779		ug/L	1	1.15	ug/L	0.068	0.2	

TriMatrix Laboratories, Inc.

Data Review Report -- Metals Lab
Sequence = 6090602 Page 4 of 4

SampleID	Analysis	IResult	Qualifier	IUnits	Dilu	FResult	FUnits	IMDL	IMRL	EMR
0808367-02	Cu Total 6020	2.761238	ME004	ug/L	1	14	ug/L	0.322	4	4
0808367-02	Ag Total 6020	0.020862		ug/L	1	0.10	ug/L	0.0726	0.2	0.2
0808367-03	V Total 6020	0.947258		ug/L	1	4.7	ug/L	0.259	4	4
0808367-03	Cu Total 6020	0.817287		ug/L	1	4.1	ug/L	0.322	4	4
0808367-03	Ag Total 6020	0.010797		ug/L	1	0.054	ug/L	0.0726	0.2	0.2
6090602-CCV4	Ag Total 6020	43.3769		ug/L	1	43.4	ug/L	0.024	0.04	
6090602-CCV4	Cu Total 6020	43.01479		ug/L	1	43.0	ug/L	0.068	0.2	
6090602-CCV4	V Total 6020	42.85077		ug/L	1	42.9	ug/L	0.17	0.2	
6090602-CCB4	Ag Total 6020	0.007124		ug/L	1	0.00712	ug/L	0.024	0.04	
6090602-CCB4	Cu Total 6020	0.007899		ug/L	1	0.00790	ug/L	0.068	0.2	
6090602-CCB4	V Total 6020	0.008801		ug/L	1	0.00880	ug/L	0.17	0.2	

UNCONTROLLED COPY

Attachment 23.5
Internal Chain Of Custody Example

24-JUL-2000 TRIMATRIX LABORATORIES, INC. PAGE #19
CHAIN OF CUSTODY REPORT FOR AN ENTIRE SUBMITTAL

CLIENT: [redacted] PROJECT: [redacted]

SUBMITTAL: Sampling Event #1 PROJECT: 35465-1

Parameter: ~~ARSENIC, TOTAL~~ All Metals DSC 7-28-00 Method: MS/EPA/WT Ref Cit: EPA-200.8/6020 Matrix: WATER

SAMPLE #	REMOVED BY: (SIGNATURE)	DATE & TIME REMOVED	RELINQUISHED BY: DATE & TIME	RECEIVED BY: DATE & TIME	DATE & TIME RETURNED
255868	Denise Coffey			7-21-00	7-28-00
255869					
255870					
255871					
255872					DSC
255873				DSC	
255874					
255875					
255876	DSC				
255877					
255878					
255879					
255880					
255881					
255882					
255883	Denise Coffey			7-21-00	7-28-00

UNCONTROLLED COPY

Attachment 23.6
SCV Standard Solution

Element	Concentration (mg/L)	Diluted Concentration (ug/L)
Aluminum	10	40
Antimony	10	40
Arsenic	10	40
Barium	10	40
Beryllium	10	40
Boron	10	40
Cadmium	10	40
Chromium	10	40
Cobalt	10	40
Copper	10	40
Lead	10	40
Manganese	10	40
Molybdenum	10	40
Nickel	10	40
Selenium	10	40
Silver	10	40
Thallium	10	40
Tin	10	40
Vanadium	10	40
Zinc	10	40
Uranium	10	40
Strontium	10	40

UNCONTROLLED COPY

Attachment 23.7
Element Conditions

Int Std	Analyte	Mass [amu]	Scan Mode	MCA Channels	Dwell Time per AMU [ms]	Integration Time [ms]	Corrections
Li		6.0151	Peak Hopping	1	100	1000	
	Be	9.0122	Peak Hopping	1	100	1000	
		44.9559	Peak Hopping	1	100	1000	
	V	50.944	Peak Hopping	1	100	1000	C10, Cr
	Cr	52.9407	Peak Hopping	1	100	1000	
	Mn	51.9405	Peak Hopping	1	100	1000	
	Co	58.9332	Peak Hopping	1	100	1000	
	+Ni	59.9332	Peak Hopping	1	100	1000	
	Ni	60.931	Peak Hopping	1	100	1000	
	+Cu	62.9298	Peak Hopping	1	100	1000	
	Cu	64.9278	Peak Hopping	1	100	1000	
	+Zn	65.926	Peak Hopping	1	100	1000	
	Zn	66.9271	Peak Hopping	1	100	1000	
	Zn	67.9249	Peak Hopping	1	100	1000	
	As	74.9216	Peak Hopping	1	100	1000	ArCl, Se
	Se	81.9767	Peak Hopping	1	100	1000	
Ge		88.9054	Peak Hopping	1	100	1000	
	Mo	97.9055	Peak Hopping	1	100	1000	Ru
	+Ag	106.905	Peak Hopping	1	100	1000	
	Ag	108.905	Peak Hopping	1	100	1000	
	+Cd	110.904	Peak Hopping	1	100	1000	Mo, Pd
	Cd	113.904	Peak Hopping	1	100	1000	Sn
In		114.904	Peak Hopping	1	100	1000	Sn
	Sb	120.904	Peak Hopping	1	100	1000	
	+Sb	122.904	Peak Hopping	1	100	1000	Te
	Ba	134.906	Peak Hopping	1	100	1000	
	+Ba	136.905	Peak Hopping	1	100	1000	
Tb		158.925	Peak Hopping	1	100	1000	
	Tl	202.972	Peak Hopping	1	100	1000	
	+Tl	204.975	Peak Hopping	1	100	1000	
	Pb	207.977	Peak Hopping	1	100	1000	Pb, Pb
Bi		208.980	Peak Hopping	1	100	1000	

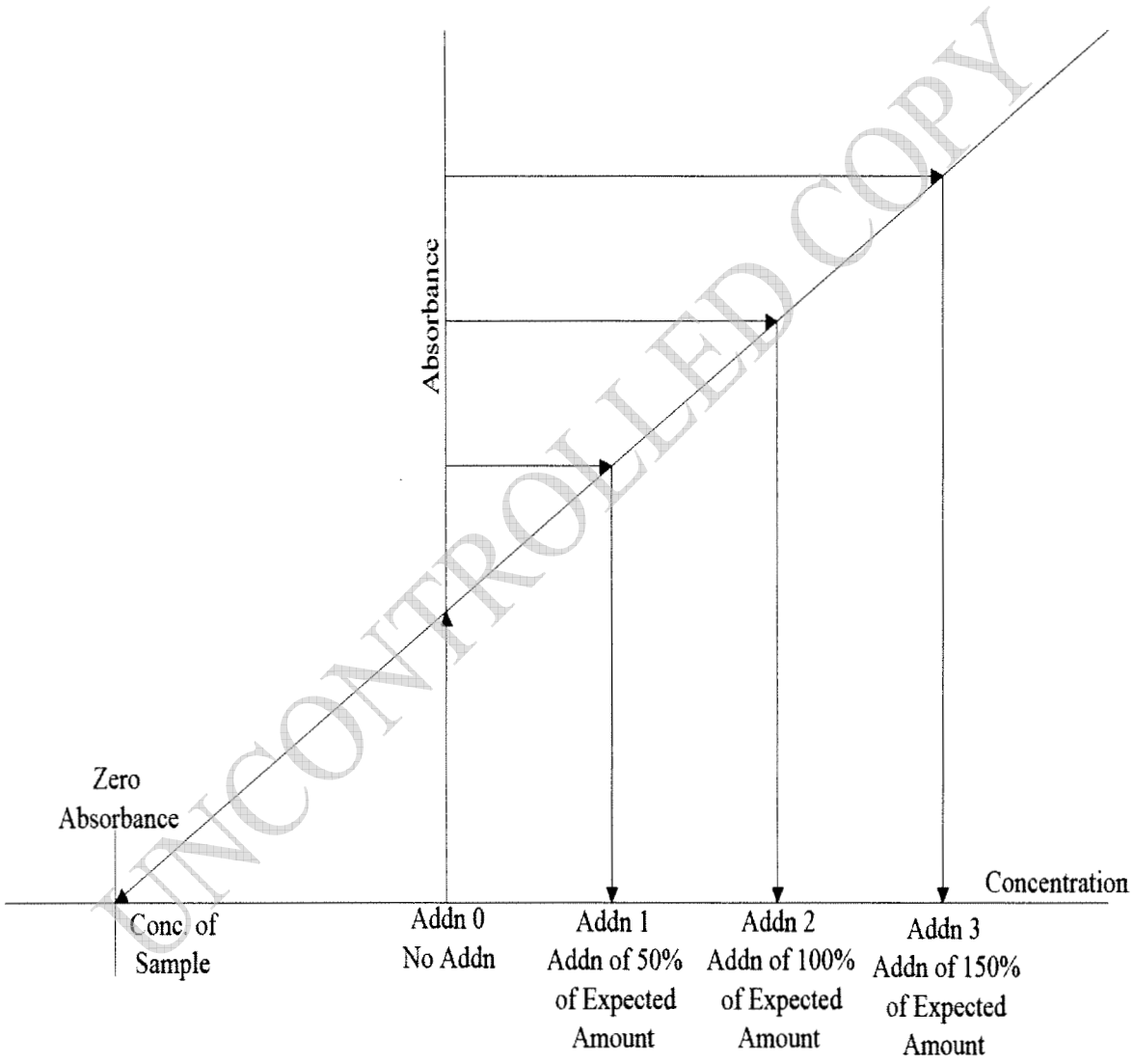
UNCONTROLLED COPY

Attachment 23.8
Spiking Solutions Final Concentrations

Element	Aqueous Samples (ug/L)	Solid Samples (mg/kg)
Aluminum	50	20
Antimony	50	20
Arsenic	50	20
Barium	50	20
Beryllium	50	20
Boron	50	20
Cadmium	50	20
Chromium	50	20
Cobalt	50	20
Copper	50	20
Lead	50	20
Manganese	50	20
Molybdenum	50	20
Nickel	50	20
Selenium	50	20
Silver	50	20
Thallium	50	20
Tin	50	20
Vanadium	50	20
Zinc	50	20

UNCONTROLLED COPY

Attachment 23.9
MSA Curve Example



UNCONTROLLED COPY

Attachment 23.10
Ionic Interference Correction Report Example

Quantitative Analysis Calibration Report

File Name:
File Path:
Calibration Type: External Calibration

Analyte	Mass	Curve Type	Slope	Intercept	Corr. Coeff.

Method Equation

Analyte	Mass	Corrections
V	50.944	-2.706684618 *(ClO 53 -(0.393948145 *Cr 52))
As	74.922	-2.706684618 *(ArCl 77 -(1.549850256 *Se 82))
Se	81.917	- 1.007833 *Kr 83
Mo	97.906	- 0.109613 *Ru 101
Cd	110.904	-0.909241101 *(MoO 108 -(0.712*Pd 106))
Cd	113.904	- 0.027250 * Sn 118
In	114.904	- 0.014038 * Sn 118
Sn	119.902	- 0.012730 * Te 125
Sb	122.904	- 0.125884 * Te 125
Pb	207.977	+1*Pb 206 +1*Pb 207

UNCONTROLLED COPY

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Block digestion of Solids for ICP and ICPMS

SW-846 Method 3050B

APPROVALS:

Area Supervisor: Marge A Scott Date: 01-15-09
Marge A. Scott

QA Officer: Date: 1/15/09
Tom C. Boocher

Operations Manager: Date: 1/15/09
Jeff P. Glaser

Procedure Number: GR-01-137
Revision Number: 1.5

Date Initiated: 2/06/01
Effective Date: 2/1/09

Date Revised: 1/15/09
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 18

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the block digestion of metals in sediment, sludge, soil and RCRA waste for ICP and ICP-MS analysis.

1.2 As the digestion does not attack silicate-bound metals, the procedure is not a total digestion technique for many samples.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 2, December 1996, Method 3050B, "Acid Digestion of Sediments, Sludge and Soils"

2.1.1 This procedure uses a 50 mL non-constricted block digestion cup as a deviation from method 3050B, which specifies a 250 mL block digestion vessel.

2.1.2 This procedure describes the use of 0.5 g dry weight as a deviation from method 3050B, which specifies the use of 1.0 g dry weight for digestion. However, acid ratios are maintained.

2.1.3 This procedure describes a routine use of the extra digestion step for antimony, tin, silicon and sometimes silver as a deviation from method 3050B, which specifies an optional digestion step for improved antimony, silver, barium and lead recovery.

3.0 SUMMARY OF PROCEDURE

3.1 Homogeneous and dried sediment, sludge and soil sample is weighed directly into a block digestion cup.

3.2 Samples are then digested with repeated additions of nitric acid then hydrogen peroxide.

3.3 If digesting for ICP, hydrochloric acid is used. If digesting for ICPMS, hydrochloric acid is not used.

3.4 All digestates are filtered then brought to a final volume of 50 mL.

3.5 Antimony, tin, silicon and sometimes silver are digested as follows:

3.5.1 Homogeneous and dried sediment, sludge and soil sample is weighed directly into a block digestion cup.

3.5.2 Samples are then digested with nitric acid and hydrochloric acid.

3.5.3 All digestates are filtered then the residue is washed with hot hydrochloric acid and hot laboratory reagent water to increase recovery.

4.0 PARAMETER OR COMPOUND LIST

4.1 This procedure includes digestion for the following elements and instrumental techniques:

ICP	
Aluminum	Silicon
Calcium	Sodium
Iron	Strontium
Lithium	Titanium
Magnesium	
Potassium	

ICP or ICPMS			
Antimony	Chromium	Nickel	Zinc
Arsenic	Cobalt	Selenium	Gold
Barium	Copper	Silver	
Beryllium	Lead	Thallium	
Boron	Manganese	Tin	
Cadmium	Molybdenum	Vanadium	

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-01-100, *Inductively Coupled Plasma Atomic Emission Spectrometry using Perkin Elmer Optima 3000/3300DV*, latest revision

5.3 TriMatrix SOP GR-01-129, *Inductively Coupled Plasma-Mass Spectrometry Perkin Elmer ELAN-6000/6100*, latest revision

5.4 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

5.5 TriMatrix SOP GR-01-115, *Mixing, Grinding and Drying Soils for Metals Analysis*, latest revision

5.6 TriMatrix SOP GR-10-113, *Laboratory Balance Calibration and Verification*, latest revision

5.7 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision

5.8 TriMatrix SOP GR-16-103, *Glassware Cleaning and Preparation for Wet Chemistry and Metals Laboratories*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Refer to TriMatrix SOP GR-01-100 for a list of interferences and associated corrective actions when digesting for ICP analysis.

6.2 Refer to TriMatrix SOP GR-01-129 for a list of interferences and associated corrective actions when digesting for ICPMS analysis.

6.3 Some sludge matrices may not be applicable to this procedure.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Hot concentrated acids are used in standard and sample preparations. Wear disposable gloves, laboratory coats and approved safety glasses at all times when digesting samples and/or working in the laboratory.

7.3 Refer to the Material Safety Data Sheet (MSDS) library for specific information on any chemical used in the laboratory.

7.4 Check fume hoods to be sure each is functioning properly. All digestions must be done in a fume hood. If the hood is not working properly, immediately contact the area supervisor. Under no circumstances is any block digestion to be performed if the exhaust hood is not on and/or functioning properly.

7.5 Many chemicals and elements used are toxic. Minimize exposure by appropriate use of the personal protective equipment provided.

7.6 No food or drink is allowed in the digestion laboratory at any time, including gum.

7.7 Wash hands before starting work. Substances on the skin may interfere with metals analysis. Wash hands after finishing work. Chemical residuals can be injested and/or carried out of the laboratory if not removed.

7.8 Before leaving the digestion laboratory, remove disposable gloves and laboratory coat. Chemical and/or acid residue transport outside the digestions laboratory needs to be kept to a minimum.

7.9 Rinse all micropipette tips and beakers before disposal or cleaning.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are typically received by the laboratory in 500 mL sample jars.

8.2 Dry at least 30 g of solid sample for analysis and quality control in accordance with TriMatrix SOP GR-01-115. Dry a larger sample aliquot to obtain at least 30 g for low-solids sludge.

8.3 The maximum holding time for all preserved samples requiring this digestion is 180 days.

8.4 Digestates (solubilized samples) ready for analysis need not be refrigerated.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Micropipette, capable of delivering 250 µL

9.2 Block digester for metals digestion, Environmental Express, HotBlock™, model SC154

9.3 Block digestion cups, 50 mL, certified pre-cleaned and calibrated, Environmental Express, catalog SC475

9.4 Ribbed watchglass, disposable, Environmental Express, catalog SC505

9.5 Filtermate™, Environmental Express, catalog SC0501, for digestate filtration

9.6 Non-ribbed watchglass, disposable, SCP Science, catalog 010-500-081

9.7 Thermometers, NIST-traceable, digital, capable of reading to at least 100° C in 0.1° C increments.

9.8 Analytical balance, capable of weighing to 0.0001 g

9.9 Filter paper, Whatman no. 41 or equivalent

9.10 PTFE sieve, USS #10

9.11 Volumetric flask, 50 mL, type A

9.12 Filter funnels

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Verify and document micropipette calibration in accordance with TriMatrix SOP GR-10-111.

10.2 Clean the block digester and counters daily or if a spill occurs.

10.3 Calibrate the analytical balance in accordance with TriMatrix SOP GR-10-113.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water (ASTM Type II), MilliQ water system

11.2 Nitric acid (HNO_3), concentrated, trace metal grade

11.3 Prepare 1:1 (v/v) HNO_3 as follows:

11.3.1 Carefully measure 50 mL of HNO_3 to 50 mL of laboratory reagent water.

11.3.2 Prepare fresh daily.

11.4 Prepare 5% (v/v) HNO_3 as follows:

11.4.1 Carefully measure 5 mL of HNO_3 to 95 mL of laboratory reagent water.

11.4.2 Prepare fresh daily.

11.5 Hydrochloric acid (HCl), concentrated, trace metal grade

11.6 Hydrogen peroxide (H_2O_2), 30%, ACS grade

12.0 STANDARDS PREPARATION

12.1 Multi-element spiking solutions are purchased as commercially prepared solutions.

12.2 Refer to Attachments 20.1 and 20.2 for ICP spiking solution examples.

12.3 Refer to Attachments 20.3 and 20.4 for ICPMS spiking solution examples.

13.0 ANALYTICAL PROCEDURE

13.1 Sediment, soil and sludge digestion is as follows:

13.1.1 Preheat the block digester and adjust the temperature so 50 mL of 5% (v/v) nitric acid is maintained at 85° C. The addition of a watchglass at 85° C adds 10 degrees and maintains the digestion at 95° ±5° C while refluxing.

13.1.2 Mix and/or grind dried sample thoroughly, passing through a USS #10 sieve if necessary. For each sample, transfer 0.5 ±0.0005 g to a block digester cup. For best results, weigh sample directly into a tared digester cup at the balance. Record the actual mass used.

13.1.3 For all digestions, add 5.0 mL of 1:1 (v/v) HNO_3. Cover with a non-ribbed watchglass and reflux in the block digester at 95° ±5° C for 15 minutes. Do not boil.

13.1.4 After 15 minutes, remove from the digester to cool then measure 2.5 mL concentrated HNO_3 into each cup and cover with a non-ribbed watchglass. Reflux for 30 minutes. Repeat this step by adding 2.5 mL acid volumes and refluxing until brown fumes are no longer given off. Brown fumes indicate the digestion is not yet complete.

13.1.5 Once digestion is complete, change to a ribbed watchglass and evaporate to approximately 2.5 mL. Do not boil or let any part of the digestion cup bottom go dry. Repeat any sample digestion in which the digestion cup goes dry. Boiling should not occur if the block digester is properly adjusted.

13.1.6 When the volume is approximately 2.5 mL, remove and cool to room temperature. It is very important to have digestates at room temperature to avoid splattering when adding hydrogen peroxide. Add 1.0 mL of laboratory reagent water and 1.5 mL of 30% H_2O_2 slowly. Let any initial reaction occur without heat.

13.1.7 Reduce the block temperature by 10° C to control excess effervescence while still maintaining enough heat to maintain the peroxide reaction. Cover with a non-ribbed watchglass and return to the block digester, heating until any reaction stops. If effervescence becomes so vigorous that splattering is immanent, control by lifting from the heat until it subsides. Do not let digestate overflow the digestion cup as analyte will be lost. Repeat the digestion if overflow occurs.

13.1.8 For samples that continue to react, carefully add another 1.0 mL of 30% H_2O_2. Do not add more than a total of 5.0 mL. Repeat until no further reaction occurs or until the maximum 5.0 mL volume has been added.

13.1.9 After the H_2O_2 digestion is complete, cover with a ribbed watchglass and again evaporate to 2.5 mL. Do not boil or let the bottom of any digestion cup go dry.

13.1.10 After reducing the volume, remove from the heat and cool to room temperature again. For ICP sample analysis only, add 5.0 mL of concentrated HCl. Cover with a non-ribbed watchglass and reflux at 95° ±5° C for 15 minutes. Skip this step if the digestion is for ICPMS analysis.

13.1.11 Cool to room temperature, dilute to 50 mL with laboratory reagent water then use a Filtermate™ for removing particulates.

13.1.11.1 Perform the filtration slowly with little pressure placed to the plunger.

13.1.11.2 If excessive backpressure occurs, stop filtering and let the digestate settle out before continuing. Applying too much pressure to the Filtermate™ plunger can cause the filter to fail allowing particulates into the filtrate.

13.1.11.3 Do not rinse the filter since the final volume has already been measured.

13.1.12 Samples are ready for analysis after filtering.

13.2 Sediment, soil and sludge digestion for antimony, tin, silicon and sometimes silver is as follows:

13.2.1 Preheat the block digester and adjust the temperature so 50 mL of 5% (v/v) nitric acid is maintained at 85° C. The addition of a watchglass at 85° C adds 10 degrees and maintains the digestion at 95° ±5° C while refluxing.

13.2.2 Mix and/or grind dried sample thoroughly, passing through a USS #10 sieve if necessary. For each sample, transfer 0.5 ±0.0005 g to a block digester cup. For best results, weigh sample directly into a tared digester cup at the balance. Record the actual mass used.

13.2.3 Add 1.25 mL concentrated HNO_3 and 5.0 mL concentrated HCl. Cover with a non-ribbed watchglass and heat at 95 ±5° C for 15 minutes. Do not boil.

13.2.4 After 15 minutes, filter through Whatman no. 41 filter paper, collecting the filtrate in a 50 mL volumetric flask. Wash the filter paper while still in the filter funnel with no more than 2.5 mL of hot (~95° C) concentrated HCl then with 10 mL of hot ASTM Type II water. Collect all washings in the same 50 mL volumetric flask.

13.2.4 Return the filter and residue to the digestion cup. Add 2.5 mL of hot (~95° C) concentrated HCl. Return to the block digester with a non-ribbed watchglass and heat at 95 ±5° C until the filter paper dissolves. Remove from the digester. Rinse the watchglass and digestion cup sides with laboratory reagent water. Filter again and collect the filtrate in the same 50 mL volumetric flask. Let the filtrate cool to room temperature in the flask then dilute to volume with laboratory reagent water.

13.2.5 High concentrations of metal salts may precipitate upon cooling. If precipitation occurs, do not dilute to volume. Instead, add up to 5.0 mL of concentrated HCl to dissolve the precipitate. After the precipitate dissolves, dilute to volume.

13.2.6 After diluting, digestates are ready for analysis by ICP.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Work orders are printed out by the Laboratory Information Management System (Element™) indicating which samples need digestion.

14.1.1 Initial and date any hand-written information on the work order report.

14.1.2 Organize and archive all work order forms in accordance with the TriMatrix quality assurance manual.

14.2 Completion of each sample with all quality control is entered back into Element™ and a preparation batch report is printed.

14.2.1 Enter all information associated with the digestion before printing.

14.2.2 The preparation batch report accompanies the sample digestion batch to the instrumental laboratory.

14.2.3 Initial and date any hand-written information on the preparation batch report.

14.2.4 Organize and archive all preparation batch report forms in accordance with the TriMatrix quality assurance manual.

14.3 Maintain the use of all standards and reagents in Element™ for traceability. Certificates of analysis are maintained on the laboratory intranet library for each standard and reagent used.

14.4 Analysts preparing digestion batches are responsible for correctly filling in, transferring and filing all associated paperwork. This is essential in providing the client with defensible data.

14.5 If internal chain-of-custody (CoC) is required, it is very important that CoC forms be filled in completely and correctly. Organize, transfer and/or archive CoC forms in accordance with the TrMatrix quality control manual.

14.6 Fill in block digester maintenance logbooks completely and correctly when maintenance is performed. Indicate blank areas in logbooks with a large "Z" that is dated and initialed.

14.7 Make corrections in any logbook or on any hardcopy with a single line over the mistake (not a writeover or scribble) then date and initial any new writing.

14.8 Silicon digested by this procedure must be reported and/or narrated as being "recoverable silicon" since most soil silicates are not digested by the acids used.

15.0 QUALITY ASSURANCE

15.1 At least one digestion blank (BLK) must be carried through the entire digestion to monitor contamination with each digestion batch of up to 20 samples. Prepare the blank by digesting an empty digestion cup as a sample and using the maximum acid additions and digestion steps used in the digestion batch.

15.2 At least one blank spike (BS) must be carried through the entire digestion to document recovery without matrix interference in each digestion batch of up to 20 samples. Prepare blank spikes as follows:

15.2.1 For ICP digestions, measure 5.0 mL 1:1 (v/v) HNO_3 into a digestion cup and spike with 250 uL each of standards SSW-1A and SSW-SSS-2A. Perform the digestion using the maximum acid additions and digestion steps used in the sample batch.

15.2.2 For ICPMS digestions, measure 5.0 mL 1:1 (v/v) HNO_3 into a digestion cup and spike with 100 uL each of standards HP solutions A and B. Perform the digestion using the maximum acid additions and digestion steps used in the sample batch.

15.3 At least one matrix spike (MS)/matrix spike duplicate (MSD) must be prepared for each digestion batch of up to 20 samples to monitor matrix recovery and precision. Perform a MS/MSD for each matrix being digested (sediment, soil, sludge or unknown).

15.3.1 For ICP matrix spikes, measure 0.5 g of dried sample into a digestion cup and spike with 250 uL each of standards SSW-1A and SSW-SSS-2A. Perform the digestion using the same acid additions and digestion steps used in the un-spiked sample.

15.3.2 For ICPMS matrix spikes, measure 0.5 g of dried sample into a digestion cup and spike with 100 uL each of standards HP solutions A and B. Perform the digestion using the same acid additions and digestion steps used in the un-spiked sample.

15.4 Digestates must not boil during the evaporation step. If a digestate begins to boil during evaporation, immediately remove from the block and reduce the temperature. Some boiling will be evident during the reflux step. However, vigorous boiling during reflux will diminish the water-acid azeotrope and cause loss of analyte. If a digestate begin to boils vigorously during refluxing, immediately remove from the block and reduce the temperature. If boiling during evaporation or vigorous boiling during refluxing is not immediately addressed, discard and the re-prepare the sample and/or samples affected. Monitor the block temperature closely to minimize re-preparation.

15.5 Each commercially purchased spiking solution must be used within its expiration date. If a standard or solution has expired, remove it from the laboratory for proper disposal.

15.6 If a sample evaporates to dryness, some metals will be volatilized. If any portion of the digestion cup bottom goes dry, discard the digestate and re-prepare the sample in another batch.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). Analysts responsible for sample processing by this procedure must pass an IDC study first.

16.1.1 Initial Demonstration of Capability Study

16.1.1.1 Spike four digestion cups as in a blank spike preparation. Digest the four spikes following every step in this procedure and obtain analysis results for the four blank spikes. Analysis of the spikes must also include every step in the analytical procedure. Enter all four results obtained into the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation. Average recovery must fall within the blank spike control limits found in Element™. Relative standard deviation must be less than or equal to 20%.

16.1.1.2 Alternatively, the last four of seven results used in an MDL study may be used as the IDC, if the MDL study was run exclusively by the analyst. ONLY the last four results may be used.

16.1.1.3 If either criterion is not met, locate and correct the source of any problem and repeat the study to confirm the corrective action was successful. Repeated failure however, will confirm a general problem with the procedure or techniques used. If multiple failures occur, correct the procedure or techniques used, and repeat the study.

16.1.1.4 Samples may not be processed by any analyst until an IDC study has been successfully completed. Give copies of successful IDC study spreadsheets and raw data to the quality assurance department for training documentation.

16.1.2 Continuing Demonstration of Capability Study

16.1.2.1 Annually, a Continuing Demonstration of Capability (CDC) study is required by each analyst.

16.1.2.2 A CDC may be accomplished by repeating the IDC study, by using the last four results from an MDL study, or by using four consecutive and exclusively run blank spike results obtained from routine sample analysis since the IDC study was completed. Alternatively, successful completion of an exclusively digested performance testing study result run since completing the IDC may be used.

16.1.2.3 Process CDC data using the same demonstration of capability spreadsheet and in the same manner as the IDC.

16.2 Method Detection Limit Studies

16.2.1 A Method Detection Limit (MDL) study must be performed annually for this procedure. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence the value is above zero.

16.2.2 The procedure used for an MDL study is based on the method given in the Code of Federal Regulations, Title 40, Part 136, Appendix B, latest revision.

16.2.3 Seven replicate digestions are performed using digestion cups spiked with all elements of interest at the estimated minimum reportable concentration.

16.2.4 The standard deviation of the average for each element, using all seven results is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

16.2.5 If the concentration spiked is between the calculated MDL and ≤10 times the calculated MDL, and there are no zero percent recoveries in the seven results then the MDL passes.

16.2.6 If the MDL study does not pass acceptably, it must be re-digested. If a study needs re-digested at a different concentration, the entire set of seven needs re-digested. If a study does not pass due to poor reproducibility on one result, only that one result needs re-digested. However, only one result of the seven can be re-digested without re-digesting the entire study.

16.2.7 All seven MDL study results do not need digested in the same digestion batch.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of a laboratory chemical without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 2, December 1996, Method 3050B, "Acid Digestion of Sediments, Sludge, and Soils"

20.0 ATTACHMENTS

20.1 ICP Spiking Solution SSW-1A Certificate of Analysis Example

20.2 ICP Spiking Solution SSW-SSS-2A Certificate of Analysis Example

20.3 ICP/MS Spiking Solution A Certificate of Analysis Example

20.4 ICP/MS Spiking Solution B Certificate of Analysis Example

20.5 Work Order Report Example

20.6 Preparation Batch Report Example

Attachment 20.1
ICP Spiking Solution SSW-1A Certificate of Analysis Example

INORGANIC VENTURES

CERTIFICATE OF ANALYSIS

195 Lehigh Avenue, Suite 4
Lakewood, New Jersey 08701 · USA
inorganicventures.com

tel: 800.669.6799 · 732.901.1900
fax: 732.901.1903
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34:2000 registered Certified Reference Material (CRM) Manufacturer (Certificate #883-02). The certificate is designed and the data is determined in accordance with ISO Guide 31:2000 (Reference Materials-Contents of Certificates and Labels), ISO Guide 34:2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statistical Principals."

2.0 **DESCRIPTION OF CRM** Custom Solution

Catalog No.: TRIMATRIX-SSW-1A

Lot Number: **Y-MEB188079**

Matrix: 2% HNO3(abs)

A607108 DA 7-06-06

2,500.00 µg/mL each:
Ca, K, Mg, Na,
250.00 µg/mL each:
Al, As, P, Se, Tl,
50.00 µg/mL each:
Ag, B, Ba, Cd, Co, Cr3, Cu, Fe, Li, Mn, Ni, Pb, Sr, V, Zn,
5.00 µg/mL each:
Be

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Aluminum, Al	249.8 ± 0.7 µg/mL	Arsenic, As	250.2 ± 0.8 µg/mL	Barium, Ba	50.16 ± 0.08 µg/mL
Beryllium, Be	5.004 ± 0.010 µg/mL	Boron, B	50.07 ± 0.10 µg/mL	Cadmium, Cd	50.16 ± 0.22 µg/mL
Calcium, Ca	2,500 ± 6 µg/mL	Chromium+3, Cr3	50.11 ± 0.16 µg/mL	Cobalt, Co	50.09 ± 0.12 µg/mL
Copper, Cu	50.17 ± 0.14 µg/mL	Iron, Fe	49.84 ± 0.32 µg/mL	Lead, Pb	50.17 ± 0.11 µg/mL
Lithium, Li	49.91 ± 0.09 µg/mL	Magnesium, Mg	2,501 ± 5 µg/mL	Manganese, Mn	49.97 ± 0.12 µg/mL
Nickel, Ni	50.10 ± 0.12 µg/mL	Phosphorus, P	249.5 ± 0.6 µg/mL	Potassium, K	2,500 ± 5 µg/mL
Selenium, Se	249.8 ± 0.8 µg/mL	Silver, Ag	50.01 ± 0.11 µg/mL	Sodium, Na	2,497 ± 1 µg/mL
Strontium, Sr	50.05 ± 0.11 µg/mL	Thallium, Tl	249.9 ± 0.7 µg/mL	Vanadium, V	49.98 ± 0.14 µg/mL
Zinc, Zn	50.09 ± 0.12 µg/mL				

Certified Density: 1.049 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$

Uncertainty $(\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

Approved By: PD 1-15-09 **QA Officer**

Approved By: MAS 01-15-09 **Area Supervisor**

Attachment 20.2
ICP Spiking Solution SSW-SSS-2A Certificate of Analysis Example

INORGANIC VENTURES™

CERTIFICATE OF ANALYSIS

195 Lehigh Avenue, Suite 4
Lakewood, New Jersey 08701 · USA
inorganicventures.com

tel: 800.669.6799 · 732.901.1900
fax: 732.901.1903
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34:2000 registered Certified Reference Material (CRM) Manufacturer (Certificate #883-02). The certificate is designed and the data is determined in accordance with ISO Guide 31:2000 (Reference Materials-Contents of Certificates and Labels), ISO Guide 34:2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statistical Principals."

2.0 **DESCRIPTION OF CRM** Custom Solution

Catalog No.: TRIMATRIX-SSW-SSS-2A

Lot Number: **Y-MEB201134**

Matrix: tr. HF, 2% HNO3(abs)

A6092 80
DSC 9-11-06
EXP. 10-01-07

250.00 µg/mL each:
Sb, Si, Sn,
50.00 µg/mL each:
Mo, Ti

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Antimony, Sb	249.1 ± 0.4 µg/mL	Molybdenum, Mo	50.02 ± 0.10 µg/mL	Silicon, Si	249.7 ± 0.8 µg/mL
Tin, Sn	250.6 ± 0.9 µg/mL	Titanium, Ti	50.14 ± 0.08 µg/mL		

Certified Density: 1.015 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

($\bar{x}$) = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 TRACEBILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS

· "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
· This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

Attachment 20.3
ICP/MS Spiking Solution A Certificate of Analysis Example

ENVIRONMENTAL EXPRESS

Certificate of Analysis

HP2079-1-1L
Solution A
Lot # 605319

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.98+%	HNO_3, 3.5%	mg/L ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C Rains

Theodore C. Rains, Ph.D.
Laboratory Director

A602735 exp 3-31-07 Rec'd 2-28-06

Exp Date: MAR 07
MSDS ATTACHED

490 Wando Park Blvd.
Mt. Pleasant, South Carolina 29464
Phone: 1.843.881.6560
Toll Free: 1.800.343.5319
FAX: 1.843.881.3964
www.environmentalexpress.com

Approved By: QA 1-15-09 **QA Officer** **Approved By:** MAS 01-15-09 **Area Supervisor**

Attachment 20.4
ICP/MS Spiking Solution B Certificate of Analysis Example

ENVIRONMENTAL EXPRESS

Certificate of Analysis

HP2079-1-1L
Solution B
Lot # 605319

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.98+%	HNO_3, 2% + Tr HF	100 mg/L ± 0.5% Antimony Silicon Titanium Molybdenum Tin

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C Rains

Theodore C. Rains, Ph.D.
Laboratory Director

A602736
exp 3-31-07
mod 2-28-06

MAR '07
Exp Date:
MSDS ATTACHED

490 Wando Park Blvd.
Mt. Pleasant, South Carolina 29464
Phone: 1.843.881.6560
Toll Free: 1.800.343.5319
FAX: 1.843.881.3964
www.environmentalexpress.com

Approved By: QD 1-15-09 **QA Officer**

Approved By: MAS 01-15-09 **Area Supervisor**

Attachment 20.5
Work Order Report Example

TriMatrix Laboratories, Inc. | WORK ORDER 0609330 | Printed: 7/11/2007 5:32:43PM

Page 1 of 6

Client: [redacted]
Project: [redacted]
Work Order: [redacted]

Project Manager: **Gary L. Wood**
Project Number: **[none]**
SDG:

Report To: [redacted]

Invoice To: [redacted]

Client Due Date: **Oct-04-06 16:00 (10 day TAT)** | Report Level: **3FL**
Date Received: Sep-20-06 12:00 25.9°C | Received By: William D. Cole
Date Logged In: Sep-21-06 16:37 | Logged In By: Kristyn N. Courtnay
W.O. Comments: *3FL*
Send cc of report with invoice to Richard Hodge.

Analysis	Lab Due Date	TAT	Expires	Analysis Comments
0609330-01 06-35 [Waste] Sampled Sep-12-06 10:00 Eastern				**RCRA 8 Metals (Total & TCLP); Report pHs**
Ag TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Ag Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
As TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
As Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Ba TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Ba Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Cd TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Cd Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Cr TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Cr Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Hg TCLP 7470A	Oct-03-06 17:00	10	Oct-10-06 10:00	
Hg Total 7471A	Oct-03-06 17:00	10	Oct-10-06 10:00	
Pb TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Pb Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Se TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Se Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
TCLP Metals Extraction	Oct-03-06 17:00	10	Sep-26-06 10:00	
0609330-02 06-36 [Waste] Sampled Sep-12-06 10:00 Eastern				**RCRA 8 Metals (Total & TCLP); Report pHs**
Ag SPLP 6010B	Jan-01-80 00:00		Sep-12-06 10:00	Added for SequenceQC in: 6092720
Ag TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Ag Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
As SPLP 6010B	Jan-01-80 00:00		Sep-12-06 10:00	Added for SequenceQC in: 6092720
As TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
As Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Ba SPLP 6010B	Jan-01-80 00:00		Sep-12-06 10:00	Added for SequenceQC in: 6092720

wko_TM_Basic.rpt

Approved By: QD 1-15-09 **QA Officer** | **Approved By:** MAS 01-15-09 **Area Supervisor**

Attachment 20.6
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0610939 Page 1 of 3

Printed: 7/11/2007 5:25:28PM

Metals, Waste, 3050B Digestion

(No Surrogate)

Batch Comments: MAS 09/25/06 &:00AM-3:30PM

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0609330	Se Total 6010B	0609330	Pb Total 6010B	0609330	Cr Total 6010B
0609330	Cd Total 6010B	0609330	Ba Total 6010B	0609330	As Total 6010B
0609330	Ag Total 6010B				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (g)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client/ QC Type*	*Extraction Comments*
0610939-BLK1		Sep-25-06 07:00	MAS	0.5	50					BLANK	
0610939-BS1		Sep-25-06 07:00	MAS	0.5	50			A607108	250	LCS	
0610939-MS1		Sep-25-06 07:00	MAS	0.5	50		0609330-02	A607108	250	MATRIX SPIKE	
0610939-MS2		Sep-25-06 07:00	MAS	0.5	50		0609330-02	A607109	250	MATRIX SPIKE	
0610939-MSD1		Sep-25-06 07:00	MAS	0.5005	50		0609330-02	A607108	250	MATRIX SPIKE DUP	
0610939-MSD2		Sep-25-06 07:00	MAS	0.5005	50		0609330-02	A607109	250	MATRIX SPIKE DUP	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-03	A	Sep-25-06 07:00	MAS	0.4995	50					[redacted]	
0609330-03	A	Sep-25-06 07:00	MAS	0.4995	50					[redacted]	

Comments:	Analyst Initials:

bch_TriMatrix.rpt

Approved By: DN 1-15-09 **QA Officer**

Approved By: MAS 01-15-09 **Area Supervisor**

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Block Digestion of Mercury in Soil, Waste and Oil

SW-846 Method 7471A

UNCONTROLLED COPY

APPROVALS:

Area Supervisor: Marge A Scott Date: 07-31-09
Marge A. Scott

QA Officer: Date: 7-31-09
Tom C. Boocher

Operations Manager: Jeff P Glaser Date: 8/3/09
Jeff P. Glaser

Procedure Number: GR-01-139
Revision Number: 0.2

Date Initiated: 5/19/03
Effective Date: 8/15/09

Date Revised: 7/31/09
Pages Revised: All

By: Marge A. Scott
Total Number of Pages: 15

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure outlines the preparation of total mercury (inorganic and organic) in soil, waste and oil samples. All samples analyzed must undergo this digestion before analysis.

2.0 PRINCIPAL METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition.* Final Update III, Revision 1, September 1994, Method 7471A, "Mercury in Solid or Semi-Solid Waste (Manual Cold-Vapor Technique)"

3.0 SUMMARY OF PROCEDURE

3.1 A measured sample mass is block-digested for two minutes with aqua regia. The digestion breaks down organo-mercury compounds into mercury in the Hg^{2+} state.

4.0 PARAMETER OR COMPOUND LIST

4.1 Mercury

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal,* latest revision

5.2 TriMatrix SOP GR-09-128, *Homogenization, Grinding and Drying of Solid Samples*, latest revision

5.3 TriMatrix SOP GR-01-123, *Mercury in Aqueous and Solid Samples, Atomic Absorbance, semi-Automated Cold Vapor*, latest revision

5.4 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Soil samples often contain mercury at low concentration. When soils are mixed and ground, the possibility that liquid samples may become cross-contaminated exists and/or cause a health hazard. Therefore, soils must be mixed and ground in the fume hood and the soil preparation area must be kept free of dust and dirt.

6.2 If a mercury thermometer is broken anywhere in the laboratory, the metals lab must be notified.

UNCONTROLLED COPY

6.3 Mercury may be lost if the block digestor is hotter than 95° C. Digestions must be temperature monitored with a NIST-verified thermometer and the temperature recorded on the pretreatment benchsheet. If sample digestion occurs above 95° C, the sample batch must be re-digested. Refer to Attachment 20.2 for an example preparation batch report.

6.4 Reagents used in this procedure will become contaminated over time. If contamination is suspected in a reagent before the established expiration, the reagent must be discarded and fresh made.

6.5 Potassium permanganate is added to eliminate possible interference from sulfide as sodium sulfide.

6.6 Copper has been reported to interfere at concentrations above 10 mg/L in the digestate.

6.7 Samples high in chlorides and volatile organic materials require additional permanganate which must also be added to standards and quality control.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Concentrated acids are used in the preparation of standards and samples for mercury analysis. Gloves, labcoats and safety glasses must be worn at all times when handling concentrated acids. Gloves must also be worn when handling digested samples. Refer to the material safety data sheet (MSDS) for information on these or other chemicals utilized in this procedure.

7.3 Mercury exists in many forms and is toxic in a variety of ways. Mercury vapor is toxic if inhaled. Do not operate the block digestor if it is not properly ventilated. Elemental Hg and Hg^{2+} are toxic if ingested. Always wash hands after handling the mercury solutions.

7.4 No food or drink is allowed in the metals lab. Food or drink may become contaminated with acid or metals and will be considered hazardous. Any food or drink found in the metals laboratory must be disposed of and not eaten.

7.5 Wash hands before starting work. Chemicals may be present on the skin that interferes with mercury analysis. Wash hands before leaving the metals lab as well. Chemicals and acids may be on the skin that could eventually be ingested or passed on through casual contact.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Soil, waste and oil samples must be refrigerated. The maximum holding time for non-aqueous samples for mercury is 28 days from the time of collection.

8.2 The minimum sample size for mercury analysis is 10 g to ensure enough sample volume for quality control.

UNCONTROLLED COPY

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Balance, analytical, capable of measurement to 0.0001 g

9.2 Balance, top-loading, capable of measurement to 0.1 g

9.3 Autopipettor, capable of delivering 50 - 1,000 µL

9.4 Volumetric flasks, class A, various volumes

9.5 Block digestor, Environmental Express, HotBlock™

9.6 Digestion vessels, Environmental Express, HotBlock™, cat. SC475, certified accurate to 50 ±0.2 mL

9.7 Millipore, in-line filters, Millex HPF HV 0.45 µm, PVDF 25 mm, non-sterile, cat. SLHVM25NS

9.8 Thermometer, metal-stem, digital, NIST-verified, Fisher model 14-648-12 (or equivalent), capable of reading from -50 to 150° C. Do not use a mercury thermometer.

9.9 Incubator, Boekel, model 131400, temperature range 50 - 60° C

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Verify and document the autopipetter calibration daily.

10.2 Clean the block digestor and counters daily or more frequently when minor spills occur.

10.3 Verify balance calibrations and document in the Daily Balance Calibration Logbook daily.

11.0 CHEMICALS AND REAGENTS

11.1 Acids used in standards preparation and for sample processing must be trace-metal grade or better.

11.1.1 Concentrated hydrochloric acid, trace-metal grade (HCl)

11.1.2 Concentrated nitric acid, trace-metal grade (HNO_3)

11.2 Reagent water: ASTM Type II laboratory water prepared for mercury analysis is deionized water fed into an all-glass distillation unit. The resulting distillate is immediately placed in a plastic container and capped. This is the ONLY water acceptable for use in the metals lab.

11.3 Aqua Regia

11.3.1 In a glass bottle, slowly add three volumes of concentrated HCl to one volume of concentrated HNO_3.

UNCONTROLLED COPY

Note: Aqua regia must be prepared and used only in a hood.

11.4 Potassium permanganate solution (ACS grade), 5% (w/v)

11.4.1 In a plastic 3-liter container, place 50 g $KMnO_4$ crystals. Add 1 liter metals reagent water to the container.

11.4.2 Place a stir bar in the solution and stir on a magnetic stir plate without heat until all crystals have dissolved. Use a disposable glass pipet to probe the container bottom for excess crystals. If crystals are found, keep stirring as this is nearly a saturated solution and may take several hours to prepare. This reagent must be made fresh daily.

11.4.3 Update the reagent logbook for each batch made.

11.4.4 If any permanganate solution spills, clean it up immediately with water. If a brownish stain develops on a contacted surface, remove it with a dilute solution of hydroxylamine hydrochloride.

Note: If permanganate touches exposed skin, a brownish stain will develop. Wash the excess permanganate off immediately for 15 minutes. If splashed in the eye, wash for 15 minutes and notify laboratory management. Read the MSDS before using this chemical.

11.4.5 Potassium permanganate will collect mercury if left exposed to the atmosphere. The solution must be kept in a sealed container when not in use and must be stored in a dark cabinet to reduce the potential for auto-catalytic reduction.

11.4.6 At no time must permanganate be exposed to organic materials or reducing agents outside the scope of this procedure. A fire or explosion could result. Do not let the solution dry and form crystals on any surface or in any container.

11.5 Sodium chloride/hydroxylamine hydrochloride solution, 12% (w/v)

11.5.1 In a plastic 3-liter container, place 60 g of sodium chloride and 60 g of hydroxylamine hydrochloride.

11.5.2 Add 500 mL of metals reagent water and a stir bar. Place the container on a stir plate without heat and stir until all crystals have dissolved. This reagent must be prepared fresh daily.

11.5.3 Update the reagent logbook for each batch made.

12.0 STANDARDS PREPARATION

12.1 The mercury stock solution for calibration is purchased commercially at a concentration of 1,000 mg/L. The second-source calibration verification (SCV) solution is purchased commercially at 1,000 mg/L from an alternate vender. All purchased standards are single-element solutions, certified to be NIST-traceable.

UNCONTROLLED COPY

12.1.1 Purchased standards have an expiration date of one year from the date received unless otherwise specified by the vender. Whichever is sooner.

12.1.2 When received in the laboratory, record in the Standards Logbook (Attachment 20.4), giving each standard a unique book/line/page identifier.

12.1.3 All intermediate and working standards must be replaced daily. These standards do not need recorded in the Standards Logbook. However, all preparation details and the purchased standard identifier must be recorded on the analytical benchsheet.

12.2 Prepare a 1000 ug/L intermediate solution as follows:

12.2.1 Rinse a clean 100 mL volumetric flask with metals reagent water, several times.

12.2.2 Place about 20 mL of metals reagent water in the volumetric.

12.2.3 Pipet 0.5 mL concentrated trace-metal grade HNO_3 into the volumetric and swirl.

12.2.4 Pipet 100 µL of 1,000 mg/L purchased mercury solution into the volumetric.

12.2.5 Dilute to the mark with metals reagent water. Cap and mix well.

12.2.6 Record in the standards logbook and write the book/page/line identifier on the volumetric flask.

12.3 Calibration Standards

12.3.1 Six concentrations are prepared for use in the initial calibration curve. These typically include a blank, a low-level at the reporting limit (0.2 ug/L), three mid-levels and a high-level near the upper linear range (5.0 ug/L).

12.3.1 Calibration standards are first prepared by rinsing six clean 50 mL block digestion vessels several times with metals reagent water. Label as 5.0 ug/L Hg, 2.0 ug/L Hg, 1.0 ug/L Hg, 0.5 ug/L Hg, 0.2 ug/L Hg, 0.0 ug/L Hg.

12.3.2 Add approximately 2.5 mL metals reagent water to each volumetric.

12.3.3 Pipet 0.25 mL concentrated trace-metal grade HNO_3 to each and swirl.

12.3.4 Pipet 250 µL of the 1,000 ug/L mercury standard (Section 12.2) into the 50 mL flask labeled 5.0 ug/L Hg.

12.3.5 Pipet 100 µL of the 1,000 ug/L Hg standard (Section 12.2) into the 50 mL flask labeled 2.0 ug/L Hg.

12.3.6 Pipet 50 µL of the 1,000 ug/L Hg standard (Section 12.2) into the 50 mL flask labeled 1.0 ug/L Hg.

UNCONTROLLED COPY

12.3.7 Pipet 25 µL of the 1,000 ug/L Hg standard (Section 12.2) into the 50 mL flask labeled 0.5 ug/L Hg.

12.3.8 Pipet 10 µL of the 1,000 ug/L Hg standard (Section 12.2) into the 50 mL flask labeled 0.2 ug/L Hg.

12.3.9 Add 2.5 mL metals reagent water to the flask labeled 0.0 ug/L Hg.

12.3.10 Digest in accordance with Sections 13.6 through 13.13.

12.4 Preparation of the second-source calibration verification (SCV) is as follows:

12.4.1 Prepare a second-source 1000 ug/L intermediate solution as follows:

12.4.1.1 Add about 20 mL of metals reagent water and pipet 0.5 mL concentrated HNO_3 into a 100 mL volumetric flask. Swirl to mix.

12.4.1.2 Pipet 100 µL of the secondary 1,000 mg/L stock into the flask and dilute to volume with metals reagent water.

12.4.2 Preparation of the 3.0 ug/L SCV is as follows:

12.4.2.1 Rinse a clean 50 mL block digestion vessel several times with metals reagent water and label it "SCV".

12.4.2.2 Add approximately 2.5 mL metals reagent water.

12.4.2.3 Pipet 250 µL concentrated HNO_3 into the flask and swirl.

12.4.2.4 Pipet 150 µL of the 1,000 ug/L solution (Section 12.5.1) into the vessel.

12.4.2.5 Digest in accordance with Sections 13.6 through 13.13.

12.5 Matrix Spiking

12.5.1 Samples need Matrix Spiked (MS/SPK) and Matrix Spike Duplicated (MSD/SPK) at a frequency of 5% or with each digestion batch of up to 20 samples. Prepare a separate MS/MSD for each matrix digested within a sample batch (soil, waste, oil or unknown).

12.5.1.1 Prepare two duplicate digestion vessels of sample to be spiked by following Sections 13.2 through 13.4.

12.5.1.2 Add 100 µL of 1,000 ug/L Hg (Section 12.5.1) to each vessel.

12.5.1.3 Digest in accordance with Sections 13.5 through 13.13.

UNCONTROLLED COPY

12.5.2 A Blank Spike (LFB/BS) is prepared with each digestion batch of up to 20 samples.

12.5.2.1 Prepare a blank spike by pipetting 100 µL of 1,000 ug/L Hg (Section 12.4.1) into a digestion vessel .

12.5.2.2 Place the vessel into the drying oven at 50-60° C in accordance with TriMatrix SOP GR-01-115.

12.5.2.3 Digest in accordance with Sections 13.5 through 13.13.

13.0 ANALYTICAL PROCEDURE

13.1 All calibration standards, quality control and client samples must be digested before analysis.

13.2 Rinse all digestion vessels and caps with metals reagent water before use.

13.3 Dry and homogenize all soils and solid waste before digesting according to TriMatrix SOP GR-01-115. Do not attempt to dry oil samples.

13.4 Weigh three separate aliquots of homogeneously mixed sample (dried, if not an oil) into a digestion vessel. The total of the three weighings must be 0.3000 ± 0.0005 g.

13.5 Add 2.5 mL of metals reagent water.

13.6 Pipet 2.5 mL aqua regia into the vessel.

13.7 Position a cap loosely on the vessel and heat at 90-95° C for 2 minutes in the block digestor.

13.8 Remove and let cool for 10 minutes then add 25 mL metals reagent water and 7.5 mL 5% $KMnO_4$ then let stand for 15 minutes. If a digestate does not maintain the purple or brown color associated with excess permanganate, discard it and weigh out a smaller total aliquot to digest. After cooling from the permanganate addition, screw the cap on.

13.9 Return to the block digestor and digest with the cap screwed on for 30 minutes at 90-95° C.

13.10 After digestion, let cool to room temperature and add 3.0 mL 12% $NaCl/NH_2OH$ then cap and shake. If the permanganate color does not clear, incrementally add 0.5 mL of the 12% $NaCl/NH_2OH$ solution until all permanganate color is gone.

13.11 When the permanganate color is gone, dilute to 50 mL with metals reagent water in the digestion vessel (each is calibrated to 50 mL) then cap and mix.

13.12 All digestates need shaken immediately before analysis. If a delay occurs between setting up and running, re-shake the entire batch to prevent layering. All sample and matrix control digestates (samples, digestion blanks, blank spikes and matrix spikes) need passed through a 0.45 µm filter immediately before analysis

UNCONTROLLED COPY

13.13 After filtering (when necessary) digestates are ready for analysis.

14.0 DATA REPORTING AND DELIVERABLES

14.1 The analyst is responsible for data quality and for correctly filling in the proper documentation. This is required for quality control and to provide the client with defensible data.

14.2 Laboratory information management system (LIMS) reporting is as follows:

14.2.1 When the analyst has finished a batch analysis all data must be input to Element™, the laboratory information management system. Benchsheets must be filled in completely to insure that results are reported correctly and all data is associated with the right quality control batch.

14.2.2 If internal chain-of-custody is required it is very important the COC form be filled in correctly.

14.2.3 All benchsheets (including COC forms) must be filed in the project-specific raw data folder.

14.3 Laboratory Required Paperwork

14.3.1 All logbooks must be filled in completely and correctly.

14.3.2 All corrections are to be made in indelible ink.

14.3.3 **Corrections are to be made with a single lineout which is then dated and initialed. Write-overs are not acceptable.** Each erroneous result must remain legible. The new result must be placed near the incorrect result.

14.3.4 Blank lines in logbooks must be Z'd out.

15.0 QUALITY ASSURANCE

15.1 One digestion blank (MPB/BLK) is carried through the entire digestion to monitor contamination for each analysis batch. The blank is prepared in the same way as a sample using an empty digestion vessel.

15.2 One blank spike (LFB/BS) is carried through the entire digestion to monitor recovery efficiency using an empty digestion vessel. Refer to Section 12.5.2 for a detailed description on how to prepare.

15.3 Matrix Spike/Matrix Spike Duplicates are performed at a frequency of 5% or for each batch of up to 20 samples per matrix, to monitor matrix recovery efficiency. Refer to Section 12.5.1 for a detailed description on how to prepare.

15.4 Digestions must not boil. If a digestion begins boiling, immediately remove it from the block digestor and lower the temperature. Replace the digestion at the lower temperature and continue digesting at 90-95° C.

UNCONTROLLED COPY

15.5 Ensure that the spiking solution used is within the expiration date by checking the standards log or the label. If a spiking solution has expired, do not use and prepare another.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an acceptable Initial Demonstration of Capability (IDC). Annually, a Continuing Demonstration of Capability (CDC) is required.

16.1.1 Initial Demonstration of Capability

16.1.1.1 Prepare four blank spikes at 0.417 mg/kg Hg in empty digestion vessels. The spiking solution used must be prepared independently from the calibration standards. Digest following every requirement in the procedure. Analyze the IDC study by calculating average percent recovery and standard deviation using the IDC spreadsheet located on the laboratory intranet library. Average percent recovery must fall within established blank spike quality control acceptance limits. The standard deviation must be ≤20%.

16.1.1.2 Alternatively, the last four of seven results obtained from an MDL study may be used as the IDC if the MDL study was run exclusively by the analyst. ONLY the last four of the seven results may be used.

16.1.1.3 If these IDC criteria are not met, locate and correct the source of the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure or techniques used. If this occurs, locate the problem and correct the procedure or techniques used then repeat the study.

16.1.1.4 Samples may not be processed by any analyst or on any new instrument until an IDC study has been successfully completed.

16.1.1.5 Copies of successful IDC and/or instrument validation spreadsheets and raw data must be given to Quality Assurance for documentation and generation of training forms.

16.1.2 Continuing Demonstration of Capability

16.1.2.1 Annually, a Continuing Demonstration of Capability (CDC) is required by each analyst.

16.1.2.2 The CDC may be accomplished by repeating the IDC, by using the last four results from the annual MDL study if run exclusively by the analyst or by processing four consecutive blank spikes run exclusively by the analyst during the course of routine sample digestion.

16.1.2.3 Process all CDC data using the same spreadsheet used for the IDC study.

UNCONTROLLED COPY

16.1.2.4 Alternatively, the successful completion of a Performance Testing sample analyzed exclusively by the analyst during the preceding year may be used for a CDC.

16.2 A Method Detection Limit study is required annually in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written disposal instructions, for that particular material. Absolutely no mercury is to be disposed of in the laboratory sink.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. All MSDS information is maintained on the laboratory intranet library.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September, 1994, Method 7471A, "Mercury in Solid or Semi-Solid Waste (Manual Cold-Vapor Technique)"

20.0 ATTACHMENTS

20.1 Purchased Mercury Stock Standard Certificate of Analysis

20.2 Example Preparation Batch Report

20.3 Example Analysis Sequence Report

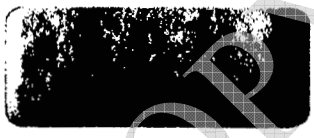
20.4 Example Stock Standards Logbook

UNCONTROLLED COPY

Attachment 20.1
Purchased Mercury Stock Standard Certificate of Analysis

[redacted]

HIGH-PURITY STANDARDS



CAT.NO.100033-1

Certificate of Analysis

Me 13.97.16
DSC
7-09-04

Mercury

Lot # 413140

Source	Source Purity	Matrix	Density g/mL @ 21.9°C	Standard Concentration
Mercury Metal	99.998%	HNO_3, 2%	1.0125	1000 ± 3 µg/mL

This spectrometric standard solution has been prepared from high-purity reference materials. Subboiled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by optical emission spectrometry and atomic absorption spectrometry

The Mercury was analyzed by ICP-MS for trace impurities. The less than values are detection limits for the method. M=Major element.

Concentrations as µg/L (ppb)

Li	<0.02	Be	<0.02	B	<1.0	Na	<1.0	Mg	<0.02	Al	<1.0	Si	<1.0	K	<1.0
Ca	<1.0	Sc	<0.02	Ti	<0.02	V	<0.02	Cr	<0.02	Mn	<0.02	Fe	<1.0	Co	<0.02
Ni	<0.02	Cu	<0.02	Zn	<0.02	Ga	<0.02	Ge	<0.02	As	<0.02	Se	<0.02	Rb	<0.02
Sr	<0.02	Y	<0.02	Zr	<0.02	Nb	<0.02	Mo	<0.02	Ru	<0.02	Rh	<0.02	Pd	<0.02
Ag	200.0	Cd	<0.02	In	<0.02	Sn	<0.02	Sb	<0.02	Te	<0.02	Cs	<0.02	Ba	<0.02
La	<0.02	Ce	<0.02	Pr	<0.02	Nd	<0.02	Sm	<0.02	Eu	<0.02	Gd	<0.02	Tb	<0.02
Dy	<0.02	Ho	<0.02	Er	<0.02	Tm	<0.02	Yb	<0.02	Lu	<0.02	Hf	<0.02	Ta	<0.02
W	<0.02	Re	<0.02	Os	<0.02	Ir	<0.02	Pt	3.0	Au	8.0	Hg	M	Tl	<0.02
Pb	<0.02	Bi	<0.02	Th	<0.02	U	<0.02								

The Standard Concentration has been certified by spectrometric analysis against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3133.

This standard is valid for eighteen months from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C Rains

Theodore C. Rains, Ph.D.
President

FEB 2006

Exp. Date

MSDS ATTACHED

UNCONTROLLED COPY

UNCONTROLLED COPY

Attachment 20.2
Example Preparation Batch Report

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0612013 Page 1 of 1

Printed: 3/30/2007 4:35:55PM

Metals, Soil, 7471A Mercury Digestion
(No Surrogate)
Batch Comments: MAS 10/16/06 2:00-2:30PM

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0610275	Hg Total 7471A	0610276	Hg Total 7471A		

Lab Number	Contain	Prepared	By	Initial (g)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0612013-BLK1		Oct-16-06 14:00	MAS	0.3	50					BLANK	
0612013-BS1		Oct-16-06 14:00	MAS	0.3	50			A606452	100	LCS	
0612013-MS1		Oct-16-06 14:00	MAS	0.3	50		0610275-03	A606452	100	MATRIX SPIKE	
0612013-MSD1		Oct-16-06 14:00	MAS	0.3003	50		0610275-03	A606452	100	MATRIX SPIKE DUP	
0610275-01	A	Oct-16-06 14:00	MAS	0.2995	50					[redacted]	
0610275-03	A	Oct-16-06 14:00	MAS	0.3003	50						
0610275-04	A	Oct-16-06 14:00	MAS	0.2999	50						
0610275-05	A	Oct-16-06 14:00	MAS	0.3003	50						
0610276-01	A	Oct-16-06 14:00	MAS	0.2995	50						
0610276-02	A	Oct-16-06 14:00	MAS	0.2998	50						
0610276-03	A	Oct-16-06 14:00	MAS	0.2996	50						
0610276-04	A	Oct-16-06 14:00	MAS	0.2997	50						
0610276-05	A	Oct-16-06 14:00	MAS	0.3005	50						
0610276-06	A	Oct-16-06 14:00	MAS	0.3005	50						
0610276-07	A	Oct-16-06 14:00	MAS	0.2999	50						
0610276-08	A	Oct-16-06 14:00	MAS	0.2998	50						
0610276-09	A	Oct-16-06 14:00	MAS	0.3005	50						

Comments	Analyst Initials

bch_TriMatrix.rpt

Approved By: [signature] 7-31-09
QA Officer

Approved By: [signature]
Area Supervisor

UNCONTROLLED COPY

Attachment 20.3
Example Analysis Sequence Report

TriMatrix Laboratories, Inc. ANALYSIS SEQUENCE **7032613** Page 1 of 2 Printed: 3/30/2007 4:53:36PM

Metals, Soil, Mar-23-07

Instrument = 216, Calibration = UNASSIGNED

Sequence Analyses:
Hg Total 7471A

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID*	*Client QC Type*	*Extraction Comments*
7032613-ICV1	QC		7030390		INITIAL CAL CHECK	
7032613-SCV1	QC		6120277		SECONDARY CAL CHECK	
7032613-ICB1	QC		7030388		INITIAL CAL BLANK	
7032613-CRL1	QC		7030393		MRL CHECK	
7032613-CCV1	QC		7030390		CALIBRATION CHECK	
7032613-CCB1	QC		7030388		CALIBRATION BLANK	
0702875-BLK1	QC				BLANK	
0702875-BS1	QC				LCS	
0703212-05	Hg Total 7471A	A 07			[redacted]	From 0702697 by MAS on Mar-21-07
0702875-MS1	QC				MATRIX SPIKE	
0702875-MSD1	QC				MATRIX SPIKE DUP	
0703291-01	Hg Total 7471A	A 07			[redacted]	soil
0702875-MS2	QC				MATRIX SPIKE	
0702875-MSD2	QC				MATRIX SPIKE DUP	
0702874-BLK1	QC				BLANK	
0702874-BS1	QC				LCS	
7032613-CCV2	QC		7030390		CALIBRATION CHECK	
7032613-CCB2	QC		7030388		CALIBRATION BLANK	
0703320-01	Hg Total 7471A	A 02			[redacted]	soil
0702874-MS1	QC				MATRIX SPIKE	
0702874-MSD1	QC				MATRIX SPIKE DUP	
0703320-02	Hg Total 7471A	A 02			[redacted]	soil
0703320-03	Hg Total 7471A	A 02			[redacted]	soil

Comments:

Analyst Initials:

seq_TriMatrix.rpt

Approved By: 7-31-09
QA Officer

Approved By: Ma Scott
Area Supervisor

UNCONTROLLED COPY

Attachment 20.4
Example Stock Standards Logbook

TriMatrix Laboratories, Inc.

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/ Volume	Solvent Used/ Lot #	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires	Math Check By
1	ME12.25-1	50	Me11-43.1 Me11-43.2	—	4/1/04	100 mg/L	50 mL	Me12.24.19	100 mL	50 mg/L	JMS	4/14/03	4/19/03	
2	ME12.25-2	20	↓	—	↓	↓	20 mL	↓	↓	20	↓	↓	↓	
3	ME12.25-3	10	↓	—	↓	↓	10 mL	↓	↓	10	↓	↓	↓	
4	ME12.25-4	1.0	Me12.24.13	—	4/19/03	~~10 mg/L~~ 1000 ppb	1 mL	↓	↓	1.0	↓	↓	↓	
5	ME12.25-5	~~4/14/03 SMK 0.04~~ 0.2	↓	—	↓	↓	200 mL	↓	↓	0.2	↓	↓	↓	
6	ME12.25-6	0.04	↓	—	↓	↓	40 mL	↓	↓	0.04	↓	↓	↓	
7	ME12.25-7	LCS	Me2 11.12	—	4/14/03	~~100 mg/L~~ 1500 ppb	400 mL	↓	↓	40	↓	↓	↓	
8	ME12.25-8	Hg ICS	Me11.4.18 HNO3	CE 226003 F 1102100	4/01/03	1000 ug/mL conc	1.0 mL 0.5 mL	ASTM II ↓	100 mL ↓	10,000 ppb 0.5%	MSS ↓	04/14/03 ↓	04/12/03 ↓	
9	ME12.25-9				03/10/03									
10	ME12.25-10	Hg	Me 10.4.13 HNO3	IV J:HG03030 F 1102100	10/01/03 03/10/03	992 ug/mL conc	1.0 mL 0.5 mL	ASTM II ↓	100 mL ↓	10,000 ppb 0.5%	MSS ↓	04/14/03 ↓	04/10/03 ↓	
11	ME12.25-11													
12	ME12.25-12													
13	ME12.25-13	Icpmssgk 19A	Me10.3.15	IV HMEB 130121	8/01/03	135 ug/mL	4.0 mL	NSTM II	100 mL	54 ug/mL	DSC	04/14/03	03/01/03	
14	ME12.25-14	Icpms 305 5A	Me10.3.14	IV HMEB 130120	08/01/03	↓	↓	↓	↓	↓	↓	↓	↓	
15	ME12.25-15	Hg	See 234711 Mod 04/14/03	CE 234711	04/01/03	—	—	—	—		MSS	04/14/03	04/14/03	
16	ME12.25-16	Hg	Me 11.4.18 HNO3	CE 226003 F 1102100	11/01/03 3/14/03	1000 ug/mL conc	1.0 mL 0.5 mL	ASTM II ↓	100 mL ↓	10,000 ppb 0.5%	MSS ↓	04/14/03 ↓	04/15/03 ↓	
17	ME12.25-17													
18	ME12.25-18													

Approved By: _____ 7-31-09
QA Officer

Approved By: _____
Area Supervisor

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Digestion of Mercury in Water, Wastewater and Aqueous Waste

EPA Method 245.1
SW-846 Method 7470A

APPROVALS:

Area Supervisor: Marge A. Scott (Marge A. Scott) Date: 01-23-09

QA Officer: (Tom C. Boocher) Date: 1-22-9

Operations Manager: Jeff P. Glaser (Jeff P. Glaser) Date: 1/23/09

Procedure Number: GR-01-140
Revision Number: 0.4

Date Initiated: 2/19/03
Effective Date: 2/20/09

Date Revised: 1/22/09
Pages Revised: All

By: Marge A. Scott
Total Number of Pages: 19

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the digestion of total mercury (inorganic and organic) in samples of groundwater, potable water, surface water, saline water, mobility leachate, and in aqueous domestic and industrial waste.

1.2 The minimum reporting limit is 0.2 ug/L.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"

2.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994

3.0 SUMMARY OF PROCEDURE

3.1 Prior to analysis, all client samples and quality control must be digested to convert organo-mercury complexes to inorganic mercury.

3.2 A measured sample aliquot, acids and potassium permanganate-potassium persulfate are transferred to a block digestion vessel and refluxed for 2 hours at 90-95° C.

3.3 The digestate is then prepared for analysis by semi-automated cold vapor atomic absorption spectrometry with the addition of hydroxylamine hydrochloride to reduce excess permanganate.

3.4 Inorganic mercury is converted to mercury in the Hg^{2+} state during the digestion, for detection and quantitation.

4.0 PARAMETER OR COMPOUND LIST

4.1 Mercury

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision

5.3 TriMatrix SOP GR-01-123, *Mercury in Aqueous and Solid Samples by Semi-Automated Cold Vapor Atomic Absorption Spectrometry*, latest revision

5.4 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Action*, latest revision

5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Soil often contains mercury at low levels. When soil samples are mixed and ground, liquid samples may become contaminated. Therefore, keep the preparation area for aqueous mercury samples free of dust and dirt to avoid cross-contamination.

6.2 Also, areas where Kjeldahl nitrogen (TKN) and/or chemical oxygen demand (COD) samples are being prepared and analyzed can be sources of contamination if not properly coordinated.

6.3 If a mercury thermometer is broken anywhere in the laboratory, notify the metals laboratory.

6.4 Mercury may be lost if the block digestor temperature exceeds 95° C. Use a digital thermometer to monitor block digestion temperature, to prevent mercury loss from boiling. Record block digestor temperatures on the pre-treatment benchsheet.

6.5 Reagents may become contaminated over time. If contamination is suspected before a reagent's assigned expiration date, discard it and acquire or prepare fresh reagent.

6.6 Volatile materials such as chlorine which absorb at 253.7 nm are a positive interference. If volatile gases are suspect, notify the instrumental analyst to purge the dead air space in each digestion cup with an inert gas before adding stannous chloride solution.

6.7 Sulfide, chloride, copper (>10 mg/L) and tellurium are interferences.

6.8 Organic compounds which absorb around 253.7 nm are interferences. Such interferant concentration levels are difficult to isolate and quality control must be strictly followed to minimize.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Concentrated acids and oxidizers are used in this procedure. Wear disposable gloves, approved safety glasses and appropriate protective clothing at all times when handling these and other reagents. Always wear disposable gloves when handling digestates as well. Refer to the MSDS library on the laboratory intranet library for information on these or other chemicals utilized in the laboratory.

7.3 Mercury exists in many forms and is toxic in a variety of ways. Mercury vapor is toxic if inhaled. Do not operate any instrument if it is not properly vented. Mercury can also be absorbed through the skin. Wear

disposable gloves at all times when handling laboratory chemicals. Elemental Hg and Hg^{2+} are toxic if ingested. Always wash hands after handling mercury or any other chemical used in the laboratory.

7.4 No food or drink is allowed in the metals laboratory or any other laboratory within the facility. Food and drink may be consumed in laboratory offices if separated from the laboratory but may only be stored in the facility cafeteria.

7.5 Wash hands before starting work. Chemicals may be present on the skin that can interfere with mercury analysis. Wash hands before leaving the metals laboratory. Chemicals and acids may be on the skin that could eventually be ingested or passed to a third party through casual contact.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples must be acidified at the time of collection to a pH of <2. If the pH is not <2 when received by the laboratory, lower to <2 during the sample receipt process using 1:1 nitric acid/water. The maximum volumes of acid added to a sample (including initial volume) are:

Container Size	Acid Volume (mL)
125	1.2
250	2.6
500	5.0
1000	10.0

NOTE: Sample preservation must occur in the original container to prevent the loss of any analytes adsorbed to the container wall. It is not permissible to remove and acidify a sample aliquot.

8.2 If a pH adjustment is necessary, a minimum of 24 hours must elapse before the sample digestion process may begin.

8.3 Consult the project chemist for instruction if the pH cannot be adjusted to <2 using the specified maximum amount of acid. Samples prepared and analyzed without proper pH preservation, results must be narrated as such.

8.4 Samples may be collected in glass or plastic. Do not allow acidified sample to come into contact with metal to prevent contaminants dissolving into the sample.

8.5 To ensure sufficient sample volume for quality control and/or redigestion, collect at least 550 mL of unfiltered liquid. Smaller volumes may result in elevated reporting limits.

8.6 Properly acid-preserved samples can be held for up to 28 days prior to digestion. Refrigeration is not required for samples or digestates.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Micropipette, capable of delivering 100 to 1000 µL

9.2 HotBlock™ block digestor, Environmental Express, model SC154

9.3 Polypropylene Digestion vessels certified pre-cleaned and pre-calibrated to 50 mL, Environmental Express, catalog SC-475

9.4 Block digestor cover

9.5 Filtermate, Environmental Express, catalog SC0501

9.6 Carboy, 50 L, high density polyethylene, with spigot

9.7 Beaker, 1 L, polyethylene

9.8 Volumetric flasks, type A, glass, 100 mL

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Verify and document micropipette calibration daily. Refer to TriMatrix GR-10-111 for micropipette calibration instructions.

10.2 Clean the block digestor and counters daily, and when spills and/or splattering occur.

11.0 CHEMICALS AND REAGENTS

11.1 Concentrated nitric acid (HNO_3), trace-metal grade

11.2 Concentrated sulfuric acid (H_2SO_4), trace-metal grade

11.3 Metals laboratory reagent water

11.3.1 ASTM Type II Milli-Q system water is fed into an all-glass Barnstead distillation unit. The distillate is immediately stored in a 50 L high-density polyethylene carboy.

11.3.2 This is the only water acceptable for use in the metals laboratory and will be designated as metals laboratory reagent water.

11.4 Potassium persulfate ($K_2S_2O_8$), ACS grade or equivalent, prepare a 5% (w/v) solution as follows:

11.4.1 Weigh out 25 g ACS-grade $K_2S_2O_8$ into a 1 L polyethylene bottle. Add 500 ml distilled reagent water to the beaker. Place a stir bar in the solution, cover and stir on a magnetic stir plate until all crystals have dissolved.

11.4.2 This solution takes several hours to prepare. Keep the reagent in a tightly capped bottle when not in use to avoid contamination. Prepare fresh weekly or sooner if a precipitate forms.

11.5 Potassium permanganate ($KMnO_4$), low-mercury ACS grade, prepare a 5% solution (w/v) as follows:

11.5.1 Weigh out and transfer 25 g $KMnO_4$ crystals to a 1 L polyethylene bottle. Add 500 ml metals laboratory reagent water to the container. Place a stir bar in the beaker then cover and stir on a magnetic stir plate until all crystals are dissolved. Since this solution is nearly saturated it may take several hours to prepare. Prepare fresh daily.

11.5.2 Update the standard number in Element™ when a new lot number of $KMnO_4$ is used in the preparation.

11.5.3 If $KMnO_4$ solution is spilled, immediately soak up with absorbent pads or paper towels then clean with water. Place all contaminated pads or towels in a plastic bag to be appropriately disposed of. If a brownish stain remains from the spill, carefully remove by applying hydroxylamine hydrochloride. Prevent exposure to the skin and eyes at all times by wearing disposable gloves, safety glasses and a laboratory coat.

11.5.4 Potassium permanganate absorbs mercury when exposed to laboratory air. Keep the reagent bottle tightly sealed when not in use. Store in a dark cabinet to minimize autocatalytic reduction.

11.6 Sodium chloride (NaCl)/hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$), prepare a 12% solution (w/v) as follows:

11.6.1 Weigh out and transfer 30 g of NaCl and 30 g of $NH_2OH{\cdot}HCl$ to a 500 mL polyethylene bottle. Add 250 mL of distilled reagent water and a stir bar.

11.6.2 Place the container on a stir plate and stir until all crystals are dissolved. Prepare fresh daily.

11.6.3 Update the standard number in Element™ when a new lot number of either NaCl or $NH_2OH{\cdot}HCl$ is used in the preparation.

12.0 STANDARDS PREPARATION

12.1 Mercury stock standard is purchased commercially at 1000 mg/L from Inorganic Ventures. The second-source secondary calibration verification (SCV) standard is purchased at 1,000 mg/L from SCP Science.

12.1.1 Purchased mercury solutions have an expiration date of the date specified by the manufacturer.

12.1.2 Log each standard into Element™.

12.1.3 All stock standards are single-element solutions.

12.2 Intermediate standard (1000 ug/L mercury)

12.2 Intermediate standard (1000 ug/L mercury)

12.2.1 Rinse a clean 100 mL volumetric flask with metals laboratory reagent water several times.

12.2.2 Add about 20 mL of metals laboratory reagent water to the flask.

12.2.3 Add 0.5 mL concentrated trace-metal grade HNO_3 then swirl to mix.

12.2.4 Pipette 100 µL of 1000 mg/L mercury standard into the flask.

12.2.5 Dilute to the volumetric mark with metals laboratory reagent water, cap tightly and mix well.

12.2.5 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation.

12.3 Prepare calibration standards as follows:

12.3.1 Prepare 0 ug/L (a blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 2.0 ug/L, and 5.0 ug/L mercury standards. Begin the preparation by labeling six 100 mL volumetric flasks. Rinse the flasks several times with metals laboratory reagent water.

12.3.2 Add approximately 20 mL metals laboratory reagent water to each flask.

12.3.3 Add 0.5 mL concentrated trace-metal grade HNO_3 to each flask and swirl.

12.3.4 Pipette 500 µL of 1000ug/L Hg standard from Section 12.3 into the vessel labeled "5.0 ug/L Hg". Dilute to volume with metals laboratory reagent water then cap and mix.

12.3.5 Pipette 200 µL of 1000 ug/L Hg standard from Section 12.3 into the vessel labeled "2.0 ug/L Hg". Dilute to volume with metals laboratory reagent water then cap and mix.

12.3.6 Pipette 100 µL of 1000 ug/L Hg standard from Section 12.3 into the vessel labeled "1.0 ug/L Hg". Dilute to volume with metals laboratory reagent water then cap and mix.

12.3.7 Pipette 50 µL of the 1000 ug/L Hg standard from Section 12.3 into the vessel labeled "0.5 ug/L Hg". Dilute to volume with metals laboratory water then cap and mix.

12.3.8 Pipette 20 µL of the 1000 ug/L Hg standard from Section 12.3 into the vessel labeled "0.2 ug/L Hg". Dilute to volume with metals laboratory water then cap and mix.

12.3.9 Dilute the volumetric labeled "Hg blank" to volume with metals laboratory water then cap and mix.

12.9.10 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation.

12.4 Prepare the Secondary Source Verification (SCV) as follows:

12.4.1 Intermediate SCV standard (1000 ug/L mercury)

12.4.1.1 Rinse a clean 100 mL volumetric flask with metals laboratory reagent water several times.

12.4.1.2 Add about 20 mL of metals laboratory reagent water to the flask.

12.4.1.3 Add 0.5 mL concentrated trace-metal grade HNO_3 then swirl to mix.

12.4.1.4 Pipette 100 µL of 1000 mg/L second-source mercury standard into the flask.

12.4.1.5 Dilute to the volumetric mark with metals laboratory reagent water, cap tightly and mix well.

12.4.1.6 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation

12.4.2 Prepare the secondary calibration verification (SCV) standard as follows:

12.4.2.1 Label a 100 mL volumetric flask as "3.0 ug/L SCV Hg". Rinse the flask several times with metals laboratory reagent water.

12.4.2.2 Add approximately 20 mL metals laboratory reagent water.

12.4.2.3 Add 0.5 mL concentrated trace-metal grade HNO_3 to each flask and swirl.

12.4.2.4 Pipette 0.30 mL of 1000 ug/L Hg standard from Section 12.4.1 into the volumetric. Dilute to volume with metals laboratory reagent water then cap and mix.

12.4.2.5 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation

12.5 Complete preparation of the calibration and SCV standards as follows:

12.5.1 Measure 30 mL of each calibration standard and the SCV into digestion vessels.

12.5.2 Pipette 0.75 mL of concentrated HNO_3 into each vessel.

12.5.3 Pipette 1.5 mL of concentrated H_2SO_4 into each vessel.

12.5.4 Swirl to mix thoroughly.

12.5.5 Pipette 4.5 mL of 5% $KMnO_4$ into each vessel and let stand for 15 minutes.

12.5.6 Pipette 2.4 mL of 5% $K_2S_2O_8$ into each vessel.

12.5.8 Place in the block digestor and position a cap on each digestion vessel without tightening then reflux for two hours at 90-95° C.

12.5.7 After digesting, cool to room temperature then pipette 1.80 mL of 12% $NaCl/NH_2OH{\cdot}HCl$ into each digestion vessel.

12.5.9 After adding, bring back to 50 mL with metals laboratory reagent water then cap tightly and mix thoroughly.

12.5.10 After a delay of at least 30 seconds, calibration standards and SCV are ready for the stannous chloride addition and immediate analysis.

12.6 Spike samples for quality control as follows:

12.6.1 Perform a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) at a frequency of at least every 10 samples for each wastewater and/or potable water matrix run in accordance with method 245.1. Perform a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) at a frequency of at least every 20 samples for each water matrix run in accordance with method 7470A..

12.6.2 Prepare 2.0 ug/L matrix spikes (MS/MSD) as follows:

12.6.2.1 Measure 30 mL of the sample being spiked into each of two block digestion vessels.

12.6.2.2 Pipette 60 µL of the 1000 ug/L spiking standard (Section 12.4.1) into each vessel.

12.6.2.3 Prepare these spiked sample aliquots in the same way the un-spiked sample is prepared.

12.6.3 Prepare a 0.5 ug/L blank spike (BS) as follows:

12.6.3.1 Measure 30 mL of metals laboratory reagent water into a block digestion vessel.

12.6.3.2 Pipette 15 µL of the 1000 ug/L spiking standard (Section 12.4.1) into the vessel.

12.6.3.3 Prepare in the same way as a sample, bringing the final volume to 50 mL.

12.6.4 Prepare a 2.0 ug/L blank spike (BS) as follows:

12.6.4.1 Measure 30 mL of metals laboratory reagent water into a block digestion vessel.

12.6.4.2 Pipette 60 µL of the 1000 ug/L spiking standard (Section 12.4.1) into the vessel.

12.6.4.3 Prepare in the same way as a sample bringing the final volume to 50 mL.

13.0 ANALYTICAL PROCEDURE

13.1 All samples and quality control must be digested before analysis. All digestates must be evaporated to below 50 mL within the digestion time and brought back to a final volume of 50 mL with metal laboratory reagent water at the end of the digestion.

13.2 Pre-heat the block digestor to 90-95° C.

13.3 Measure 30 mL of each well-mixed sample into a digestion vessel. Prepare the associated quality control samples and calibration standards.

13.4 To each digestion vessel add the following reagents:

13.4.1 Pipette in 0.75 mL of concentrated HNO_3

13.4.2 Pipette in 1.5 mL concentrated H_2SO_4

13.4.3 Pipette in 4.5 mL of $KMnO_4$

13.5 After adding, swirl to mix then let the samples sit for 15 minutes.

13.5.1 If a sample does not maintain the purple or brown permanganate color for at least 15 minutes, add up to an additional 3.0 mL of $KMnO_4$ solution but no more.

Note: Separate calibration standards and quality control must be prepared with the same permanganate volume to match the sample.

13.5.2 If a sample does not maintain the purple or brown permanganate color after the maximum $KMnO_4$ addition of 7.5 mL, discard the preparation, dilute the sample appropriately and prepare 30 mL of the dilution for digestion.

13.6 Pipette 2.40 mL of 5% $K_2S_2O_8$ into each digestion.

13.7 Place in the block digestor and position a cap on each digestion vessel without tightening then reflux for two hours at 90-95° C. Do not exceed 95° C.

13.8 After digestion, remove and cool to room temperature then pipette in 1.8 mL of 12% $NaCl/NH_2OH{\cdot}HCl$.

13.9 Finally, bring to a final volume of 50 mL with metals laboratory reagent water. Cap and mix thoroughly.

13.10 After a delay of at least 30 seconds, samples are ready for the stannous chloride addition and immediate analysis.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Work orders are printed out by the Laboratory Information Management System (Element™) indicating which samples need digestion.

14.1.1 Initial and date any hand-written information on the work order report.

14.1.2 Organize and archive all work order forms in accordance with the TriMatrix quality assurance manual.

14.2 Completion of each sample with all quality control is entered back into Element™ and a preparation batch benchsheet report is printed.

14.2.1 Enter all information associated with the digestion before printing.

14.2.2 The preparation batch report accompanies the sample digestion batch to the instrumental laboratory.

14.2.3 Initial and date any hand-written information on the preparation batch report.

14.2.4 Organize and archive all preparation batch report forms in accordance with the TriMatrix quality assurance manual.

14.3 Maintain the use of all standards and reagents in Element™ for traceability. Certificates of analysis are maintained on the laboratory intranet library for each standard and reagent used. (Attachment 20.1)

14.4 Analysts preparing digestion batches are responsible for correctly filling in, transferring and filing all associated paperwork. This is essential in providing the client with defensible data. (Attachment 20.3)

14.5 If internal chain-of-custody (COC) is required, it is very important that COC forms be filled in completely and correctly. Organize, transfer and/or archive COC forms in accordance with the TrMatrix quality control manual.

14.6 Fill in block digestor maintenance logbooks completely and correctly when maintenance is performed. Indicate blank areas in logbooks with a large "Z" that is dated and initialed.

14.7 Make corrections in any logbook or on any hardcopy with a single line over the mistake (not a writeover or scribble) then date and initial any new writing.

15.0 QUALITY ASSURANCE

15.1 Prepare and digest a method blank (BLK) for each batch of up to 20 samples digested, to monitor contamination. The BLK is 30 mL of metals laboratory reagent water measured into a block digestion vessel. The BLK vessel is then processed as a sample following every step in the procedure. Refer to TriMatrix SOP GR-01-123 for BLK acceptance criteria.

15.2 Prepare and digest the two blank spikes (BS) of Section 12.6.3 and 12.6.4 for each batch of up to 20 samples digested. Refer to TriMatrix SOP GR-01-123 for BS acceptance criteria.

15.3 Prepare and digest a matrix spike (MS)/matrix spike duplicate (MSD) as described in Section 12.6.2 and at the frequency described in Section 12.6.1, or prepare for each digestion session/matrix. Matrix spikes monitor matrix recovery and precision. Refer to TriMatrix SOP GR-01-123 for MS/MSD acceptance criteria.

15.4 Do not allow samples to boil during digestion. If a sample begins to boil, immediately remove it from the block digestor and turn the temperature down.

15.5 Do not use expired spiking solutions and/or standards. If a standard has expired, obtain or prepare another.

15.6 The method of standard additions must be performed on all leachates with 50% or less spike recovery. Refer to TriMatrix SOP GR-01-123 for preparing and digesting a standard addition series.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate an ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. A Continuing Demonstration of Capability (CDC) study is also required annually.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Prepare four blank spikes (BS) at a concentration of 2.0 ug/L in laboratory reagent water. ONLY the last four results may be used. Otherwise, digest and evaluate the four blank spikes following every step in the digestion and analysis procedures. Calculate average percent recovery and relative standard deviation by inputting the four result values to the IDC spreadsheet on the laboratory intranet library. Average percent recovery must fall within 85 – 115% recovery. Relative standard deviation must be $\leq$20%.

19.1.1.2 If either criterion is not met, locate and correct the source of the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure or techniques used. If this occurs, locate and correct the procedure or techniques used then repeat the study.

19.1.1.3 Samples may not be analyzed by any analyst until a demonstration of capability study has been successfully completed. Copies of successful study spreadsheets must be given to the quality assurance department.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A demonstration of capability must be repeated annually by all analysts running samples.

19.1.2.2 The CDC may be accomplished by repeating the IDC study, by submitting four consecutive blanks spike values obtained during the course of routine sample

digestion, by submitting the last four MDL study values or by submitting an acceptable performance testing result. In each case, the work must be done exclusively by one analyst and submitted to quality control for documentation.

19.2 A Method Detection Limit (MDL) Study must be completed annually in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) on the laboratory intranet library when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"

19.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994

20.0 ATTACHMENTS

20.1 Mercury Stock Standard Certificate of Analysis Example

20.2 Element™ Work Order Report Example

20.3 Element™ Preparation Batch Report Example

20.4 Stock Standards Log Example

20.5 Digestion Flowchart

Attachment 20.1
Mercury Stock Standard Certificate of Analysis Example

inorganic ventures / iv labs

195 lehigh avenue, suite 4, lakewood, nj 08701 usa
phone: 800-669-6799 • 732-901-1900 • fax: 732-901-1903
e-mail: ivsales@ivstandards.com • website: www.ivstandards.com

certificate of analysis

1.0 **Inorganic Ventures / IV Labs is an ISO Guide 34-2000 Certified Reference Material (CRM) Manufacturer: Certificate #883-02.** The certificate is designed and the certified value(s) and uncertainty(ies) are determined in accordance with ISO Guide 31-2000 (Reference Materials - Contents of certificates and label(s), ISO Guide 34-2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statisical Principles."

2.0 **DESCRIPTION OF CRM** Custom-Grade **1000 µg/mL Mercury in 3.5% (abs) HNO_3**

Catalog Number:	CGHG1-1, CGHG1-2, and CGHG1-5
Lot Number:	**Y-HG02038**
Starting Material:	Hg metal
Starting Material Purity (%):	99.999555
Starting Material Lot No	05214TX
Matrix:	3.5% (abs) HNO_3

Hg 14.98.8
A503277
ORR
9-2705

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 1007 ± 3 µg/mL

Certified Density: 1.026 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

Certified Value ($\bar{x}$) = $\frac{\Sigma x_i}{n}$ — ($\bar{x}$) = mean; x_i = individual results; n = number of measurements

Uncertainty (±) = $2\left[\frac{\Sigma s_i^2}{(n)}\right]^{1/2}$ — Σs_i = The summation of all significant estimated errors. (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

."Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)

. This IV product is Traceable to NIST via direct comparison to NIST SRMs. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors.

4.1 **Assay Method #1** **998 ± 4 µg/mL**
EDTA NIST SRM 928 Lot Number: 392110

Assay Method #2 **1007 ± 3 µg/mL**
ICP Assay NIST SRM 3133 Lot Number: 991304

Approved By: _____ 1-22-09 (QA Officer) Approved By: MAS 01-23-09 (Area Supervisor)

Attachment 20.2
Element™ Work Order Report Example

TriMatrix Laboratories, Inc.

WORK ORDER 0610156

Printed: 10/12/2006 2:46:21PM

Page 1 of 2

Metals Sample Receipt Notice

Client: [redacted]
Project: [redacted]
Client Due Date: **Oct-20-06 16:00 (10 day TAT)**
W.O. Comments: *2RL*

Project Manager: **Gary L. Wood**
Project Number: **[none]**
Report Level: **2RL**

Lab Number	Sample Name / Analysis	Matrix	Sampled Date / TAT	Expire Date	Sample Comments / Lab Due Date	Comments
[redacted]						
	Ag Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	As Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Ba Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Cd Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Cr Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Cu Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Hg Total 7470A		10	Nov-03-06 14:00	Oct-19-06 17:00	
	Ni Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Pb Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Se Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Zn Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	

Attachment 20.3
Element™ Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0901183 Page 1 of 1

Printed: 1/22/2009 1:38:35PM

Metals, Water, 7470A Digestion - Total

(No Surrogate)

Batch Comments: MAS 01/21/09 1:0.0-3:00PM

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0901199	Hg Total 7470A				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (mL)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0901183-BLK1		Jan-21-09 13:00	MAS	30	30					BLANK	
0901183-BS1		Jan-21-09 13:00	MAS	30	30			8100309	60	LCS	
0901183-MS1		Jan-21-09 13:00	MAS	30	30		0901199-12	8100309	60	MATRIX SPIKE	
0901183-MSD1		Jan-21-09 13:00	MAS	30	30		0901199-12	8100309	60	MATRIX SPIKE DUP	
0901199-10	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-11	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-12	E	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-13	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-14	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-15	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-16	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-17	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr

Approved By: [signature] 1-22-09 **QA Officer**

Approved By: MAS 01-23-09 **Area Supervisor**

Attachment 20.4
Stock Standards Log Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

8060501

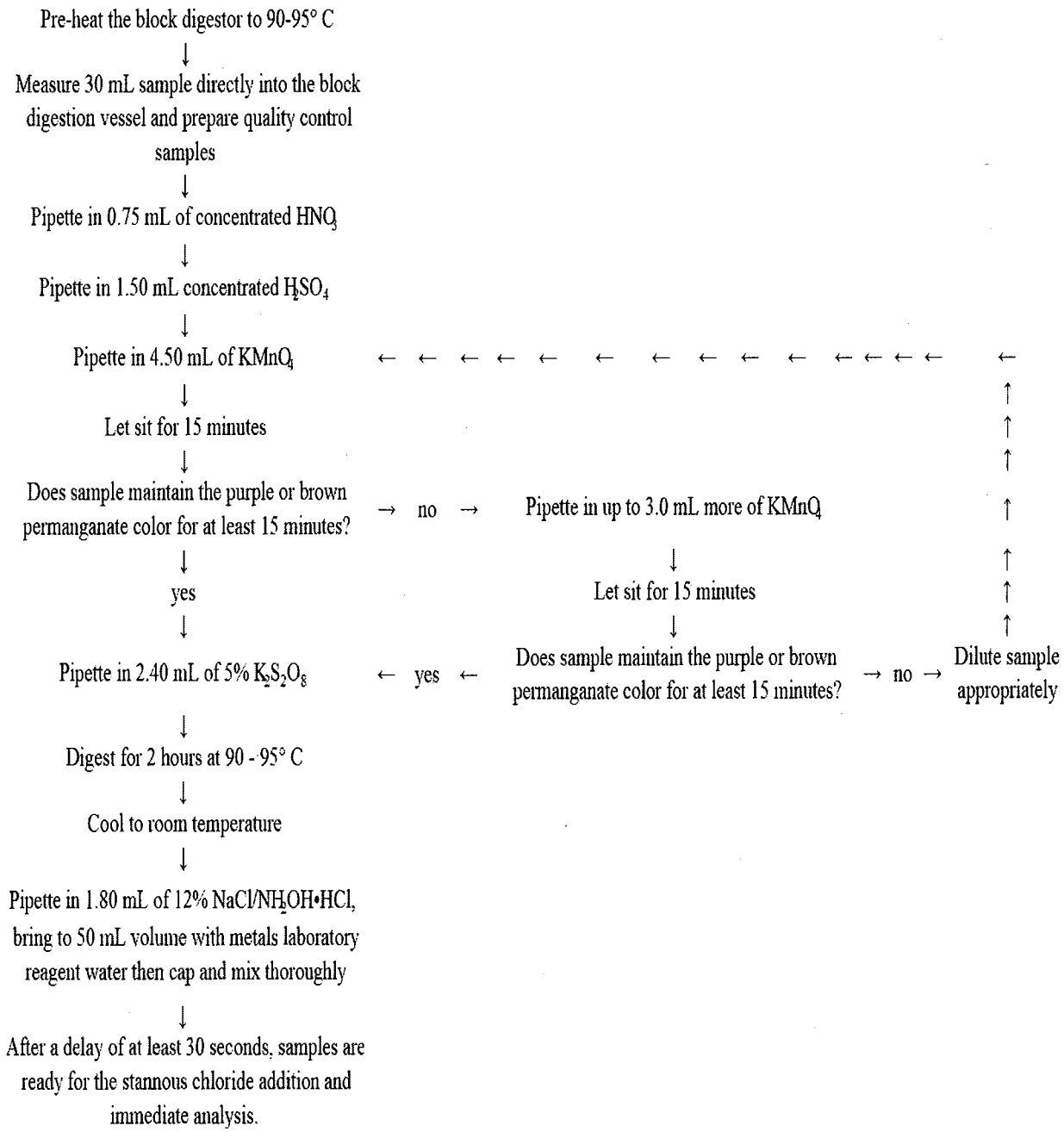
Description:	Mercury 1000 mg/L SCV Stock	Expires:	Feb-28-10
Standard Type:	Other	Prepared:	Jun-17-08
Solvent:	10% HNO3	Prepared By:	** Vendor **
Final Volume (mls):	100	Department:	Metals
Vials:	1	Last Edit:	Jun-17-08 12:56 by RGJ

SCP part# 140-051-801
Lot#SC8122815
Received 06/17/08

Analyte	CAS Number	Concentration	Units
Mercury, OWEP-TAR	7439-97-6	1000	mg/L
Mercury, OWEP-SAR	7439-97-6	1000	mg/L
Mercury	7439-97-6	1000	mg/L

Approved By: QA 1-22-09 **QA Officer** **Approved By:** MAS 01-23-09 **Area Supervisor**

Attachment 20.5
Digestion Flowchart



Approved By: [signature] 1-22-09 **QA Officer**

Approved By: MAS 01-23-09 **Area Supervisor**

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Block Digestion of Total Metals in Water for ICP

EPA Method 200.7
SW-846 Method 3010A

APPROVALS:

Area Supervisor: Marge A Scott Date: 01/14/09
Marge A. Scott

QA Officer: _____ Date: 1-8-09
Tom C. Boocher

Operations Manager: _____ Date: 1/15/09
Jeff P. Glaser

Procedure Number: GR-01-147
Revision Number: 0.4

Date Initiated: 7/15/03
Effective Date: 1/20/09

Date Revised: 1/7/09
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 16

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 Water, wastewater and leachate samples are prepared for total metals analysis by block digestion.

1.2 Silica-based matrices are not digested using this procedure.

1.3 Digestates are prepared for analysis by inductively coupled plasma spectrometry (ICP).

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 1, July 1992, Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy"

2.1.1 This procedure describes the use of a 50 mL non-constricted block digestion cup as a deviation from method 3010A, which specifies the use of a Griffin beaker.

2.1.2 This procedure describes the use of 25 mL as a deviation from method 3010A, which specifies a 100 mL sample aliquot for digestion.

2.1.3 This procedure describes the use of 5 mL of nitric acid for the first reflux as a deviation from method 3010A which specifies 3 mL. Acid ratios are maintained and 1.25 mL is used for 25 mL instead of 5 mL for 100 mL.

2.2 *Code of Federal Regulations*, Title 40, Appendix C to Part 136, February 1, 2002, Method 200.7, "Inductively Coupled Plasma-Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes"

2.2.1 This procedure describes the use of a 50 mL block non-constricted digestion cup as a deviation from method 200.7, which specifies the use of a Griffin beaker.

3.0 SUMMARY OF PROCEDURE

3.1 Samples are prepared as follows:

3.1.1 A 25.0 mL aliquot of well-mixed, homogeneous aqueous sample is accurately measured into a digestion cup.

3.1.2 Analytes are solubilized by evaporating the sample to low volume after adding nitric acid while gently refluxing. Additional portions of nitric acid are added with further refluxing until the digestate is light in color or has color-stabilized.

3.1.3 The digestate is then gently refluxed with 1:1 (v/v) hydrochloric acid to dissolve any precipitate.

3.1.4 After digestion is complete and cool, the volume is adjusted with laboratory reagent water to 25.0 mL.

4.0 PARAMETER OR COMPOUND LIST

4.1 This procedure includes digestion for the following elements:

Aluminum	Chromium	Selenium	Calcium	Potassium
Antimony	Cobalt	Silver	Iron	Silicon
Arsenic	Copper	Strontium	Lithium	Sodium
Barium	Lead	Thallium	Magnesium	Sulfur
Beryllium	Manganese	Tin	Phosphorus	Titanium
Boron	Molybdenum	Vanadium		
Cadmium	Nickel	Zinc		

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-01-100, *Inductively Coupled Plasma Atomic Emission Spectrometry using Perkin Elmer Optima 3000/3300DV*, latest revision

5.3 TriMatrix SOP GR-01-145, *Inductively Coupled Plasma Atomic Emission Spectrometry using Perkin Elmer Optima 3000/3300DV*, latest revision

5.4 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Refer to TriMatrix SOP GR-01-145 for a list of interferences and associated corrective actions when digesting for ICP analysis.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Concentrated acids are used in standard and sample preparations. Wear disposable gloves, laboratory coats and approved safety glasses at all times when digesting samples and/or working in the laboratory.

7.3 Refer to the Material Safety Data Sheet (MSDS) library for specific information on any chemical used in the laboratory.

7.4 Check fume hoods to be sure each is functioning properly. All digestions must be done in a fume hood. If the hood is not working properly, immediately contact the area supervisor. Under no circumstances is any block digestion to be performed if the exhaust hood is not function properly.

7.5 Many chemicals and elements used in this procedure are toxic. Minimize exposure by appropriate use of the personal protective equipment provided.

7.6 No food or drink is allowed in the digestion laboratory at any time, including gum.

7.7 Wash hands before starting work. Substances on the skin may interfere with metals analysis. Wash hands after finishing work. Chemical residuals can be injested and/or carried out of the laboratory if not removed.

7.8 Before leaving the digestion laboratory, remove disposable gloves and laboratory coat. Chemical and/or acid residue transport outside the digestions laboratory needs to be kept to a minimum.

7.9 Rinse all micropipette tips and beakers before disposal or cleaning.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND MATERIALS

8.1 Samples collected for digestion and analysis by ICP can be preserved in the field or upon receipt by the laboratory. If the pH is not <2 when received by the laboratory, lower to <2 during the sample receipt process using 1:1 nitric acid/water. The maximum volumes of acid added to a sample (including initial volume) are:

Container Size	Acid Volume (mL)
125	1.2
250	2.6
500	5.0
1000	10.0

8.2 If a pH adjustment is necessary, a minimum of 24 hours must elapse before the sample digestion process may begin.

NOTE: Sample preservation must occur in the original container to prevent the loss of any analytes adsorbed to the container wall. It is not permissible to remove and acidify a sample aliquot.

NOTE: If the sample must be analyzed within 24 hours of collection, the sample must be preserved immediately during collection.

NOTE: If boron is an analyte of interest the sample must be preserved immediately during collection.

NOTE: Samples collected for pre-treatment and analysis by method 6010 must be preserved immediately during collection.

8.3 Consult the project chemist for instruction if the pH cannot be adjusted to <2 using the specified maximum amount of acid. Samples prepared and analyzed without proper pH preservation, results must be narrated as such.

8.4 Samples may be collected in glass or plastic. If Boron is to be analyzed, use only plastic containers. Do not allow acidified sample to come into contact with metal to prevent contaminants dissolving into the sample.

8.5 To ensure sufficient sample volume for quality control and/or redigestion, collect at least 150 mL of unfiltered liquid. Smaller volumes may result in elevated reporting limits.

8.6 For some determinations of dissolved elements, the filtered and preserved sample may require digestion. Filtered samples that form a precipitate at preservation, during transit to the laboratory or in storage must be digested before the analysis.

8.7 Properly acid-preserved samples can be held for up to 180 days prior to digestion. Refrigeration is not required for samples or digestates.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Micropipette, capable of delivering 125 µL

9.2 Block digestor for metals digestion, Environmental Express, HotBlock™, model SC154

9.3 Block digestion cups, certified pre-cleaned, catalog SC475, available from Environmental Express

9.4 Ribbed watchglasses, disposable, Environmental Express, catalog SC505

9.5 Filtermate, Environmental Express, catalog SC0501, for digestate filtration

9.6 Non-ribbed watchglasses, disposable, Environmental Express, SCP Science, catalog 010-500-081

9.7 Thermometers, NIST-traceable, digital, capable of reading to at least 100° C in 0.1° C increments.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Verify and document the micropipette calibration daily.

10.2 Clean each block digestor and all counters daily. Clean up spills immediately.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water (ASTM Type II), milliQ water system

11.2 Nitric acid (HNO_3), concentrated, trace metal grade

11.3 Hydrochloric acid (HCl), concentrated, trace metal grade

11.3.1 Prepare a 1:1 (v/v) solution of HCl in water by measuring 50 mL of concentrated HCl into 50 mL of laboratory reagent water. Remember to perform the addition in a fume hood.

11.3.2 Expiration is six months from the date made.

12.0 STANDARDS PREPARATION

12.1 Multi-element ICP spiking solutions are purchased as commercially prepared solutions. Refer to Attachments 20.1 and 20.2 for ICP spiking solution examples.

13.0 ANALYTICAL PROCEDURE

13.1 Fill one block digestion cup with laboratory reagent water and place in a random hole of the block digestor. Cover with a watchglass that has a hole in the center and insert the digital thermometer through the hole. Using the thermometer and not the block digestor read-out, adjust the block temperature to 90 - 95° C.

13.2 Mix samples by vigorously shaking to achieve homogeneity. Immediately after shaking, measure 25.0 mL (or an appropriately smaller volume) into a block digestion cup. For samples requiring matrix spikes, shake vigorously before each 25.0 mL aliquot is removed.

13.3 Measure 0.75 mL of concentrated HNO_3 into each sample cup then cover with a ribbed watchglass.

13.4 After the block equilibrates to 90 - 95° C, place all samples in the block and cover with a ribbed watchglass. Evaporate to near dryness, which is approximately 1.25 mL. If necessary, measure 1.25 mL into an empty digestion cup for comparison. If any digestion goes to dryness, discard and re-prepare with another sample aliquot.

13.5 After evaporating to near dryness, cool by removing briefly from the block then add 1.25 mL of concentrated HNO_3. Cover with a non-ribbed watchglass then return to the block and gently reflux. Slight boiling should occur while refluxing but vigorous boiling must be avoided. Continue refluxing. If the digestate is not light in color after refluxing, cool and add another 0.75 mL HNO_3 then continue refluxing as before. Repeat 0.75 mL HNO_3 additions and refluxing until the digestate is light in color or does not change with continued refluxing.

13.6 When the digestion is complete, change to a ribbed watchglass and evaporate to approximately 0.75 mL. Be careful not to let any digestion go to dryness, else it will need discarded and re-prepared.

13.7 After evaporation to 0.75 mL, remove from the block to cool then measure 2.5 mL of 1:1 HCl into each digestate. Return to the block and gently reflux for an additional 15 minutes.

13.8 After refluxing with HCl, remove from the block and cool to room temperature.

13.9 When cool, dilute to 25.0 mL with laboratory reagent water then cap and shake vigorously. At this point, digestates are ready for analysis.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Work orders are printed out by the Laboratory Information Management System (Element™) indicating which samples need digestion.

14.1.1 Initial and date any hand-written information on the work order report.

14.1.2 Organize and archive all work order forms in accordance with the TriMatrix quality assurance manual.

14.2 Completion of each sample with all quality control is entered back into Element™ and a preparation batch report is printed.

14.2.1 Enter all information associated with the digestion before printing.

14.2.2 The preparation batch report accompanies the sample digestion batch to the instrumental laboratory.

14.2.3 Initial and date any hand-written information on the preparation batch report.

14.2.4 Organize and archive all preparation batch report forms in accordance with the TriMatrix quality assurance manual.

14.3 Maintain the use of all standards and reagents in Element™ for traceability. Certificates of analysis are maintained on the laboratory intranet library for each standard and reagent used.

14.4 Analysts preparing digestion batches are responsible for correctly filling in, transferring and filing all associated paperwork. This is essential in providing the client with defensible data.

14.5 If internal chain-of-custody (COC) is required, it is very important that COC forms be filled in completely and correctly. Organize, transfer and/or archive COC forms in accordance with the TrMatrix quality control manual.

14.6 Fill in block digestor maintenance logbooks completely and correctly when maintenance is performed. Indicate blank areas in logbooks with a large "Z" that is dated and initialed.

14.7 Make corrections in any logbook or on any hardcopy with a single line over the mistake (not a writeover or scribble) then date and initial any new writing.

15.0 QUALITY ASSURANCE

15.1 At least one digestion blank (BLK) must be carried through the entire digestion to monitor contamination with each digestion batch of up to 20 samples. The blank is prepared by measuring 25.0 mL of laboratory reagent water or appropriate extraction fluid into a digestion cup and digesting as a sample.

15.2 At least one blank spike (BS) must be carried through the entire digestion to document recovery without matrix interference in each digestion batch of up to 20 samples. A blank spike is prepared by measuring 25.0 mL of laboratory reagent water or appropriate extraction fluid into a digestion cup, then spiking with 125 µL of solution SSW-1A and 125 µL of solution SSW/SSS-2A.

15.3 Prepare a matrix spike (MS) and matrix spike duplicate (MSD) for water samples, for each digestion batch of up to 20 samples.

15.4 Prepare a matrix spike (MS) and matrix spike duplicate (MSD) for leachates, for each digestion batch of up to 20 samples.

15.5 Prepare a matrix spike (MS) and matrix spike duplicate (MSD) for wastewater samples, for each digestion batch of up to 10 samples.

15.6 Prepare matrix spike and matrix spike duplicates by measuring a 25.0 mL sample aliquot into a digestion cup, then spiking with 125 µL of solution SSW-1A and 125 µL of solution SSW/SSS-2A.

15.7 Digestates must not boil during the evaporation step. If a digestate begins to boil during evaporation, immediately remove from the block and reduce the temperature. Some boiling will be evident during the reflux step. However, vigorous boiling during reflux will diminish the water-acid azeotrope and cause loss of analyte. If a digestate begin to boils vigorously during refluxing, immediately remove from the block and reduce the temperature. If boiling during evaporation or vigorous boiling during refluxing is not immediately addressed, discard and the re-prepare the sample and/or samples affected. Monitor the block temperature closely to minimize re-preparation.

15.8 Each commercially purchased spiking solution must be used within its expiration date. If a standard or solution has expired, remove it from the laboratory for proper disposal.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). Analysts responsible for sample processing by this procedure must pass an IDC study first.

16.1.1 Initial Demonstration of Capability Study

16.1.1.1 Spike four 25.0 mL aliquots of laboratory reagent water as in a blank spike preparation. Digest the four spikes following every step in this procedure and obtain analysis results for the four blank spikes. Analysis of the spikes must also include every step in the analytical procedure. Enter all four results obtained into the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation. Average recovery must fall

within the blank spike control limits found in Element™. Relative standard deviation must be less than or equal to 20%.

16.1.1.2 Alternatively, the last four of seven results used in an MDL study may be used as the IDC, if the MDL study was run exclusively by the analyst. ONLY the last four results may be used.

16.1.1.3 If either criterion is not met, locate and correct the source of any problem and repeat the study to confirm the corrective action was successful. Repeated failure however, will confirm a general problem with the procedure or techniques used. If multiple failures occur, correct the procedure or techniques used, and repeat the study.

16.1.1.4 Samples may not be processed by any analyst until an IDC study has been successfully completed. Give copies of successful IDC study spreadsheets and raw data to the quality assurance department for training documentation.

16.1.2 Continuing Demonstration of Capability Study

16.1.2.1 Annually, a Continuing Demonstration of Capability (CDC) study is required by each analyst.

16.1.2.2 A CDC may be accomplished by repeating the IDC study, by using the last four results from an MDL study, or by using four consecutive and exclusively run blank spike results obtained from routine sample analysis since the IDC study was completed. Alternatively, successful completion of an exclusively digested performance testing study result run since completing the IDC may be used.

16.1.2.3 Process CDC data using the same demonstration of capability spreadsheet and in the same manner as the IDC.

16.2 Method Detection Limit Studies

16.2.1 A Method Detection Limit (MDL) study must be performed annually for this procedure. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence the value is above zero.

16.2.2 The procedure used for an MDL study is based on the method given in the Code of Federal Regulations, Title 40, Part 136, Appendix B, latest revision.

16.2.3 Seven replicate digestions are performed using laboratory reagent water spiked with the elements(s) of interest at the estimated minimum reportable concentration.

16.2.4 The standard deviation of the average for each element, using all seven results is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

16.2.5 If the concentration spiked is between the calculated MDL and ≤10 times the calculated MDL, and there are no zero percent recoveries in the seven results then the MDL passes.

16.2.6 If the MDL study does not pass acceptably, it must be re-digested. If a study needs re-digested at a different concentration, the entire set of seven needs re-digested. If a study does not pass due to poor reproducibility on one result, only that one result needs re-digested. However, only one result of the seven can be re-digested without re-digesting the entire study.

16.2.7 All seven MDL study results do not need digested in the same digestion batch.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of a laboratory chemical without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranent library.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 1, July 1992, Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy"

19.2 *Code of Federal Regulations*, Title 40, Appendix C to Part 136, February 1, 2002, Method 200.7, "Inductively Coupled Plasma-Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes"

20.0 ATTACHMENTS

20.1 ICP Spiking Solution SSW-1A Certificate of Analysis Example

20.2 ICP Spiking Solution SSW-SSS-2A Certificate of Analysis Example

20.3 Work Order Report Example

20.4 Preparation Batch Report Example

20.5 Digestion Flowchart

Attachment 20.1
ICP Spiking Solution SSW-1A Certificate of Analysis Example

INORGANIC VENTURES
195 Lehigh Avenue, Suite 4
Lakewood, New Jersey 08701 · USA
inorganicventures.com

CERTIFICATE OF ANALYSIS

tel: 800.669.6799 · 732.901.1900
fax: 732.901.1903
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34:2000 registered Certified Reference Material (CRM) Manufacturer (Certificate #883-02). The certificate is designed and the data is determined in accordance with ISO Guide 31:2000 (Reference Materials-Contents of Certificates and Labels), ISO Guide 34:2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statistical Principals."

2.0 **DESCRIPTION OF CRM** Custom Solution

Catalog No.: TRIMATRIX-SSW-1A

Lot Number: **Y-MEB188079**

Matrix: 2% HNO3(abs)

A607108 DA 7-06-06

2,500.00 µg/mL each:
Ca, K, Mg, Na,
250.00 µg/mL each:
Al, As, P, Se, Tl,
50.00 µg/mL each:
Ag, B, Ba, Cd, Co, Cr3, Cu, Fe, Li, Mn, Ni, Pb, Sr, V, Zn,
5.00 µg/mL each:
Be

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Aluminum, Al	249.8 ± 0.7 µg/mL	Arsenic, As	250.2 ± 0.8 µg/mL	Barium, Ba	50.16 ± 0.08 µg/mL
Beryllium, Be	5.004 ± 0.010 µg/mL	Boron, B	50.07 ± 0.10 µg/mL	Cadmium, Cd	50.16 ± 0.22 µg/mL
Calcium, Ca	2,500 ± 6 µg/mL	Chromium+3, Cr3	50.11 ± 0.16 µg/mL	Cobalt, Co	50.09 ± 0.12 µg/mL
Copper, Cu	50.17 ± 0.14 µg/mL	Iron, Fe	49.84 ± 0.32 µg/mL	Lead, Pb	50.17 ± 0.11 µg/mL
Lithium, Li	49.91 ± 0.06 µg/mL	Magnesium, Mg	2,501 ± 5 µg/mL	Manganese, Mn	49.97 ± 0.12 µg/mL
Nickel, Ni	50.10 ± 0.12 µg/mL	Phosphorus, P	249.6 ± 0.6 µg/mL	Potassium, K	2,500 ± 5 µg/mL
Selenium, Se	249.8 ± 0.8 µg/mL	Silver, Ag	50.01 ± 0.11 µg/mL	Sodium, Na	2,497 ± 1 µg/mL
Strontium, Sr	50.05 ± 0.11 µg/mL	Thallium, Tl	249.9 ± 0.7 µg/mL	Vanadium, V	49.98 ± 0.14 µg/mL
Zinc, Zn	50.09 ± 0.12 µg/mL				

Certified Density: 1.049 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

Attachment 20.2
ICP Spiking Solution SSW-SSS-2A Certificate of Analysis Example

INORGANIC VENTURES™

195 Lehigh Avenue, Suite 4
Lakewood, New Jersey 08701 · USA
inorganicventures.com

tel: 800.669.6799 · 732.901.1900
fax: 732.901.1903
info@inorganicventures.com

CERTIFICATE OF ANALYSIS

1.0 **INORGANIC VENTURES** is an ISO Guide 34:2000 registered Certified Reference Material (CRM) Manufacturer (Certificate #883-02). The certificate is designed and the data is determined in accordance with ISO Guide 31:2000 (Reference Materials-Contents of Certificates and Labels), ISO Guide 34:2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statistical Principals."

2.0 **DESCRIPTION OF CRM**

Catalog No.:	TRIMATRIX-SSW-SSS-2A	
Lot Number:	**Y-MEB201134**	
Matrix:	tr. HF,	2% HNO3(abs)

A6092S0
Rec 9-11-06
exp. 10-01-07

250.00 µg/mL each:
Sb, Si, Sn,
50.00 µg/mL each:
Mo, Ti

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Antimony, Sb	249.1 ± 0.4 µg/mL	Molybdenum, Mo	50.02 ± 0.10 µg/mL	Silicon, Si	249.7 ± 0.8 µg/mL
Tin, Sn	250.6 ± 0.9 µg/mL	Titanium, Ti	50.14 ± 0.08 µg/mL		

Certified Density: 1.015 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

($\bar{x}$) = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 TRACEBILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

Attachment 20.3
Work Order Report Example

TriMatrix Laboratories, Inc. **WORK ORDER 0609285** Printed: 9/20/2006 2:30:22PM

Page 1 of 2

Metals Sample Receipt Notice

Client: [redacted]
Project: **Analytical Services**
Client Due Date: **Oct-04-06 16:00 (10 day TAT)**
W.O. Comments: *IRL*

Project Manager: **Lisa M. Harvey**
Project Number: **32207**
Report Level: **1RL**

Lab Number	Sample Name / Analysis	Matrix	Sampled Date / TAT	Expire Date	Sample Comments / Lab Due Date	Comments
[redacted]						
	Ag Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Cd Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Cr Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Cu Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Ni Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Pb Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Zn Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	

Approved By: _____ 1-8-09 **QA Officer** **Approved By:** MAS 01/14/09 **Area Supervisor**

Attachment 20.4
Preparation Batch Report Example

TriMatrix Laboratories, Inc. **PREPARATION BATCH** 0610868 Page 1 of 2 Printed: 9/22/2006 9:36:45AM

Metals, Waste Water, 3010A Digestion

Batch comments:
09/22/06 7:00am-3:30pm
Inst 219 #179 @ 95C
Hotblock Vessel Lot #A511LS106

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	Client Source ID	Client Spike ID	Client uL Spike	Comments	
0609168-01	A	Sep-22-06 07:00	MAS	25	25	[redacted]				
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7				
0609168-03	A	Sep-22-06 07:00	MAS	25	25	[redacted]				
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7				
0609285-01	C	Sep-22-06 07:00	MAS	25	25	[redacted]				
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7				
0609295-01	D	Sep-22-06 07:00	MAS	25	25	[redacted]				
Na Total 200.7										
0609300-04	A	Sep-22-06 07:00	MAS	25	25	[redacted]			report at 10 ug/L	report at 10 ug/L
Cu Total 200.7										
0609303-01	B	Sep-22-06 07:00	MAS	25	25	[redacted]			Added for BatchQC in: 0610868	BatchQC
Ag Total 200.7	Al Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Mo Total 200.7	Na Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7	
0609304-01	B	Sep-22-06 07:00	MAS	25	25	[redacted]			wkly	wkly
Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Zn Total 200.7							
0609305-01	A	Sep-22-06 07:00	MAS	25	25	[redacted]				
Ag Total 200.7	Al Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7			
0609316-01	C	Sep-22-06 07:00	MAS	25	25	[redacted]				
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7				
0609327-02	F	Sep-22-06 07:00	MAS	25	25	[redacted]				
Mo Total 200.7										
0609327-04	F	Sep-22-06 07:00	MAS	25	25	[redacted]				
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7				
0609337-01	C	Sep-22-06 07:00	MAS	25	25	[redacted]				
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7				

Comments: | Analyst Initials:

bch_TM_byAnalysis.rpt

Approved By: [signature] 1-8-09 **QA Officer** **Approved By:** MAS 01/14/09 **Area Supervisor**

Attachment 20.5
Digestion Flowchart

Measure 25.0 mL of homogeneous sample into a digestion vessel
↓
add 0.75 mL of concentrated nitric acid
↓
cover with a ribbed watchglass
↓
evaporate to about 1.25 mL at 90-95 deg. C (do not boil)
↓
remove from the block digestor to cool then add 1.25 mL concentrated nitric acid
↓
change to a non-ribbed watchglass and return to the block digestor
↓
reflux (slight boiling will be observed but vigorous boiling must be avoided)
↓
if the digestate is not light in color after refluxing, cool and add another 0.75 mL nitric acidthen continue refluxing as before
↓
repeat 0.75 mL nitric acid additions and refluxing until the digestion is complete
↓
remove from the block digestor and change to a ribbed watchglass
↓
evaporate to about 0.75 mL at 90-95 deg. C (do not boil)
↓
remove from the block digestor to cool then add 2.5 mL 1:1 hydrochloric acid
↓
change to a non-ribbed watchglass and return to the block digestor
↓
reflux gently for 15 minutes
↓
remove from the block digestor and cool to room temperature
↓
dilute to 25.0 mL with laboratory reagent water then cap and shake vigorously
↓
digestates are ready for analysis

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Block Digestion of Aqueous Samples and Extracts for Total/Dissolved Metals by ICPMS

SW-846 Method 3020A

APPROVALS:

Area Supervisor: Marge A. Scott (Marge A. Scott) Date: 01-14-09

QA Officer: (Tom C. Boocher) Date: 1-8-09

Operations Manager: (Jeff P. Glaser) Date: 1/15/09

Procedure Number: GR-01-148
Revision Number: 0.3

Date Initiated: 12/3/01
Effective Date: 1/20/09

Date Revised: 1/7/09
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 13

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure outlines total metals digestion in water and leachate samples with the exception of silica-based matrices. Also outlined is the preparation of aqueous samples for dissolved metals analysis. A block digestion system is used when digestion is required.

1.2 Samples that have been filtered and acidified for dissolved metals analysis do not need the acid digestion unless otherwise specified.

1.2 The digestion is for analysis by inductively coupled plasma/mass spectrum (ICP/MS).

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, July 1992, Method 3020A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy"

3.0 SUMMARY OF PROCEDURE

3.1 A representative sample aliquot is refluxed in nitric acid until the solution is light in color or the color remains stable. The digestate is then diluted to a predetermined volume for analysis.

3.2 Deviation from the referenced method involves sample volume reduction from 100 to 25 mL, with nitric acid additions adjusted in direct proportion to the volume reduced.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to Attachment 20.1.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-16-103, Glassware Cleaning and Preparation for the Wet Chemistry and Metals Laboratories, latest revision

5.3 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

5.4 TriMatrix SOP GR-10-111, *Fixed and Adjustable Mechanical Pipette and Bottle Top Dispenser Calibration Verification*, latest revision

5.5 TriMatrix SOP GR-10-113, *Laboratory Balance Calibration and Verification*, latest revision

5.6 TriMatrix SOP GR-10-106, Inorganic and Metals Laboratories Corrective Actions, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Samples must be at room temperature before a digestion aliquot can be taken to assure measurement accuracy.

7.0 SAFETY PRECAUTIONS

7.1 Refer to the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan for routine precautions.

7.2 Gloves, approved safety glasses and laboratory coats must be worn at all times when handling acids and when performing this procedure. An impervious plastic apron is also required when handling significant volumes of acid.

7.3 Concentrated nitric acid fumes burn, discolor the skin and give off poisonous gases. Fume hoods must be used during handling and when performing this procedure.

7.4 Rinse all micropipette tips and beakers before cleaning or disposal. Do not dispose of acid rinses before neutralizing.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND MATERIALS

8.1 Samples must be acidified at the time of collection to a pH of <2 with nitric acid. If the pH is not <2 when received by the laboratory, lower to <2 during sample receipt using 1:1 nitric acid/water. The maximum allowable acid volumes that may be added to a sample (including initial volume) are:

Container Size (mL)	Acid Volume (mL)
125	1.2
250	2.6
500	5.0
1000	10

Note: Sample preservation must occur in the original container to prevent analyte loss from plating out on the container wall. It is not permissible to remove and acidify a sample aliquot.

8.2 If pH adjustment is necessary when received at the laboratory, a minimum of 24 hours must elapse before the sample may be digested.

8.3 Consult the project chemist for instructions if the pH cannot be adjusted to <2 using the maximum acid volume. Samples digested and analyzed without proper pH preservation, results must be narrated as such.

8.4 Samples must be collected in plastic. Do not allow acidified sample to come into contact with metal to prevent contaminants dissolving into the sample.

8.5 To ensure sufficient sample volume for quality control and/or redigestion, collect at least 500 mL of unfiltered liquid. Smaller volumes may result in elevated reporting limits.

8.6 Samples collected for dissolved metals analysis must be filtered and the filtrate acidified at the time of collection. For some sample determinations of dissolved elements, the filtered/acidified filtrate may require digestion.

8.7 Appropriately acid-preserved samples may be held for up to 180 days prior to digestion. Refrigeration is not required for samples or digestates.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Micropipetter – capable of delivering 25 and 100 uL

9.2 Block digester for metals digestion – Environmental Express HotBlock™

9.3 Digestion cups, polypropylene, certified precleaned – Environmental Express SC499 or SC500

9.4 Ribbed watch glasses – Environmental Express SC505

9.5 Filtermate (digestate filtration) – Environmental Express SC0501

Note: If digestates are filtered after digestion, all associated quality control must be filtered as well.

9.6 Nonribbed watch glasses – SCP Science 010-500-081

9.7 Filtration units, vacuum pump, for dissolved sample filtering in the field

Note: Each lot of filtration units received must be verified by running a dissolved blank and a dissolved blank spike. Results must be uploaded to Element™.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Verify and document micropipette calibration daily.

10.2 Clean block digestion units and counters daily and/or when spills occur.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory distilled, deionized water (ASTM Type II) - designated in this procedure as metals-free water

11.2 Concentrated nitric acid (HNO_3), trace metal grade

12.0 STANDARDS PREPARATION

12.1 Multi-element ICPMS certified spiking standards are listed in Attachment 20.1.

13.0 ANALYTICAL PROCEDURE

13.1 Prepare samples that have been filtered through a 0.45 µm filter and acidified in the field as follows:

13.1.1 If a filtered/acidified sample exhibits a strong organic odor, digest before analysis.

Note: Do not directly smell any sample.

13.1.2 If a filtered/acidified sample exhibits significant color, digest before analysis.

13.1.3 If a filtered/acidified sample has a turbidity greater than 1 NTU, digest before analysis.

13.1.4 If a filtered/acidified sample forms a precipitate at preservation, during transit to the laboratory or in storage, digest before analysis.

13.2 To perform the digestion, mix samples by shaking vigorously to achieve homogeneity. After mixing, measure 25.0 mL of sample (or an appropriate dilution) into a polypropylene digestion cup using the cup graduations. For samples requiring matrix spikes, shake again before subsequent 25.0 mL transfers.

Note: Each digestion cup lot needs verified for acceptable accuracy at 25 mL by weighing out 25 mL of metals-free water on the analytical balance. Then, calculate the average volume measured and uncertainty.

13.3 Add 0.75 mL concentrated HNO_3, cover each cup with a ribbed watchglass and reflux in the block digester at 90 - 95° C. Adjust the block digester temperature to 85° C before refluxing to account for the watchglass, which adds ten degrees. Refer to the HotBlock™ operating manual for temperature adjustment and principles of operation. Evaporate to near dryness which is approximately 1.25 mL but don't let any digestion go completely dry.

13.4 After reducing to about 1.25 mL, remove from the block digester and cool to room temperature.

13.5 Add 0.75 mL concentrated HNO_3, cover with a nonribbed watchglass and gently reflux for 30 minutes. Repeat with additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

13.6 When digestion is complete, evaporate to 0.75 mL using a ribbed watchglass. Do not let any part of the digestion cup bottom evaporate to dryness. If a digestate evaporates to dryness, it must be repeated.

13.7 Remove from the block digester and add 2.5 mL metals-free water. Mix and continue warming for 15 minutes.

13.8 Remove from the block digester and cool to room temperature.

13.9 After cooling, dilute to 25.0 mL with metals-free water, cap tightly and shake vigorously.

14.0 DATA REPORTING AND DELIVERABLES

14.1 All samples that require digestion and must be input as pretreated to the Laboratory Information Management System (Element™).

14.2 Each sample batch of up to 20 samples referencing the same digestion method is given an Element™ batch number.

14.2 The batch number is used to segregate digestion batches for analysis. The batch number is also used by Element™ to associate quality control with associated sample results.

15.0 QUALITY ASSURANCE

15.1 A Digestion Blank (BLK) must be carried through the entire procedure to monitor for contamination with each digestion batch of up to 20 samples. The BLK is prepared by measuring 25 mL of metals-free water or appropriate leachate extraction fluid into a digestion cup for digestion.

Note: A dissolved metals blank is metals-free water that is matrix-matched to instrument standards at the instrument.

15.2 A digestion Blank Spike (BS) must be carried through the entire procedure to monitor recovery for each digestion batch.

15.2.1 The digestion blank spike is prepared by measuring 25.0 mL of metals-free water or appropriate leachate extraction fluid into a digestion cup and spiking with 25 µL each of two 50 mg/L mixed metals solutions (refer to Attachment 20.1).

15.2.1.1 HP2079-1-1L, stock solution A (100 mg/L) is diluted to 50 mg/L and designated QCA.

15.2.1.2 HP2079-2-1L, stock solution B (100 mg/L), is diluted to 50 mg/L and designated QCB.

15.2.2 A dissolved metals blank spike is prepared in metals-free water, spiked as above then matrix-matched to the instrument standards.

15.3 Perform matrix spike/matrix spike duplicates (MS/MSD) for each digestion batch of up to 20 samples for each matrix type.

15.3.1 The digestion matrix spike is prepared by measuring 25.0 mL of sample into a digestion cup and spiking with 25 µL each of two 50 mg/L mixed metals solutions (refer to Attachment 20.1).

15.3.1.1 HP2079-1-1L, stock solution A (100 mg/L) is diluted to 50 mg/L and designated QCA.

15.3.1.2 HP2079-2-1L, stock solution B (100 mg/L), is diluted to 50 mg/L and designated QCB.

15.3.2 A dissolved metals matrix spike is prepared in filtered/acidified sample, matrix-matched to the instrument standards and spiked as above.

15.4 If a digestate evaporates to dryness, some metals will be volatized. If any portion of a digestion cup bottom goes dry, discard the digestate and redigest in another batch.

15.5 Digestates must not boil during evaporation. If a digestate begins boiling, immediately remove from the block digester and reduce the temperature setting. Excessive boiling will cause analyte loss. If a digestate boils during evaporation, it must be discarded and the sample redigested in another batch.

15.6 Standard solutions must be used within the predetermined expiration date. If a standard or solution has expired, remove it from the laboratory for proper disposal and obtain or prepare another.

15.7 Quality control acceptance limits are listed in Element™.

15.8 Address unacceptable quality control and perform corrective action in accordance with TriMatrix SOP GR-10-106.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC).

16.1.1 To perform an IDC, prepare four blank spikes by spiking 25 mL of metals-free water with 25 µL of 50 mg/L spiking standard.

16.1.2 Digest as samples, following every step in the procedure and analyze.

16.1.3 Input results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation.

16.1.4 Average recovery must fall within the blank spike control limits in Element™. Relative standard deviation must not be higher than 20%.

16.1.5 If any criterion is not met, locate and correct the source of the problem and repeat the study successfully.

16.1.6 Repeated failure however, will confirm a general problem with the procedure and/or techniques used. If multiple failures occur, correct the procedure and/or technique used, revise the procedure and repeat the study successfully.

16.1.7 Samples may not be processed by any analyst until an IDC study has been successfully completed.

16.1.8 Submit a copy of the successful IDC spreadsheet to the Quality Assurance department for training documentation.

16.2 Annually, a Continuing Demonstration of Capability (CDC) is required by each analyst by any of the following approaches:

16.2.1 By repeating the IDC study.

16.2.2 By inputting the last four results from a method detection limit (MDL) study to the IDC spreadsheet if prepared exclusively by the analyst.

16.2.3 By inputting four consecutive blank spike results obtained during the course of routine sample analysis to the IDC spreadsheet if prepared exclusively by the analyst.

16.2.4 By obtaining a passing result on a blind performance testing study if prepared exclusively by the analyst.

16.3 A method detection limit (MDL) study is required annually in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, July 1992, Method 3020A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy"

20.0 ATTACHMENTS

20.1 Analyte List

20.2 Example Preparation Batch Report

20.3 Example Environmental Express Certificate of Analysis, Solution A

20.4 Example Environmental Express Certificate of Analysis, Solution B

Attachment 20.1
Analyte List

Element	Environmental Express Stock Solution
Aluminum	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Antimony	HP2079-1-1L (QCB) 100 mg/L diluted to 50 mg/L
Arsenic	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Barium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Beryllium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Boron	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Cadmium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Chromium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Cobalt	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Copper	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Lead	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Manganese	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Nickel	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Selenium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Silver	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Strontium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Thallium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Tin	HP2079-1-1L (QCB) 100 mg/L diluted to 50 mg/L
Vanadium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Zinc	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L

Attachment 20.2
Example Preparation Batch Report

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0808883 Page 1 of 1

Printed: 8/11/2008 2:04:12PM

Metals, Water, 3020A Digestion

Batch comments:
MAS 07/30/08 7:35AM-3:30PM
INST#217 @95C#195
HOTBLOCK VESSEL LOT#A801LP001

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	Client Source ID	Client Spike ID	Client uL Spike	Comments
0807506-01	O	Jul-30-08 07:35	MAS	25	125	[redacted]			
Ag Total 6020	*As Total 6020*	*B Total 6020*	*Ba Total 6020*	*Be Total 6020*	*Cd Total 6020*	*Co Total 6020*	*Cr Total 6020*		
Cu Total 6020	*Ni Total 6020*	*Pb Total 6020*	*Sb Total 6020*	*Se Total 6020*	*Tl Total 6020*	*V Total 6020*	*Zn Total 6020*		
0807506-02	E	Jul-30-08 07:35	MAS	25	125	[redacted]			
Ag Total 6020	*As Total 6020*	*B Total 6020*	*Ba Total 6020*	*Be Total 6020*	*Cd Total 6020*	*Co Total 6020*	*Cr Total 6020*		
Cu Total 6020	*Ni Total 6020*	*Pb Total 6020*	*Sb Total 6020*	*Se Total 6020*	*Tl Total 6020*	*V Total 6020*	*Zn Total 6020*		
0808883-BLK1		Jul-30-08 07:35	MAS	25	125				
0808883-BS1		Jul-30-08 07:35	MAS	25	125		8070952	25	
0808883-MS1		Jul-30-08 07:35	MAS	25	125	0807506-02	8070952	25	
0808883-MSD1		Jul-30-08 07:35	MAS	25	125	0807506-02	8070952	25	
0808883-PS1		Aug-04-08 09:40	DSC	2	10	0807506-02	7110622	50	

Comments:	Analyst Initials:

bch_TM_byAnalysis.rpt

Approved By: QA 1-8-09 **QA Officer**

Approved By: MAS 01-14-09 **Area Supervisor**

Attachment 20.3
Example Environmental Express Certificate of Analysis, Solution A

Me 14.62.4
A 505048

ENVIRONMENTAL EXPRESS

Certificate of Analysis

HP2079-1-1L
SOLUTION A
Lot # 511212

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts, or Oxides	99.99+%	HNO_3, 3.5%	mg/L ± 0.5% See elements listed on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Subboiled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by optical emission spectrometry and atomic absorption spectrometry.

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures.

The Standard Concentration has been certified by spectrometric analysis against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series, and checked by ICP prior to shipping.

This solution is valid for a period of one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains, Ph.D.
Laboratory Director

Exp. Date MAY 06
MSDS ATTACHED

490 Wando Park Blvd.
Mt. Pleasant, South Carolina 29464
Phone: 1.843.881.6560
Toll Free: 1.800.343.5319
FAX: 1.843.881.3964
www.environmentalexpress.com

Approved By: QA 1-8-09 QA Officer
Approved By: MAS 01-14-09 Area Supervisor

Attachment 20.4
Example Environmental Express Certificate of Analysis, Solution B

ENVIRONMENTAL EXPRESS

Certificate of Analysis

HP2079-1-1L
Solution B
Lot # 631157

6110557
DSL
11-15-08
EXP. 12-01-09

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.98+%	HNO_3, 2% + Tr HF	100 mg/L ± 0.5% Antimony Silicon from $(NH_4)_2SiF_6$ Titanium Molybdenum Tin

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains, Ph.D.
Laboratory Director

Exp Date: DEC '07
MSDS ATTACHED

490 Wando Park Blvd.
Mt. Pleasant, South Carolina 29464
Phone: 1.843.881.6560
Toll Free: 1.800.343.5319
FAX: 1.843.881.3964
www.environmentalexpress.com

TRIMATRIX LABORATORIES

STANDARD OPERATING PROCEDURE

Organochlorine Pesticides Analysis by Gas Chromatography (GC)

EPA Methods 608 and 608.2
SW-846 Method 8081B

UNCONTROLLED COPY

APPROVALS:

Area Supervisor: Jodi L. Blouw Date: 2-10-10

QA Officer: Tom C. Boocher Date: 1-29-10

Operations Manager: Jeff P. Glaser Date: 2/10/10

Procedure Number: GR-03-120
Revision Number: 4.5

Date Initiated: 10/12/94
Effective Date: 2/10/10

Date Revised: 1/29/10
Pages Revised: All

By: Daniel J. Mierendorf
Total Number of Pages: 32

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure outlines the analysis of organochlorine pesticides. Direct inject gas chromatography (GC) applicable to components listed in Attachments 23.1 and 23.2 (Tables 1 and 1A) is used for characterization of groundwater, soil, sludge, water-miscible and non-water-miscible waste. Other analytes may be determined, provided each is analyzed in accordance with all procedural guidelines.

1.2 Use a confirmatory column of dissimilar polarity or gas chromatography/mass spectrometry (GCMS) to verify all positive results.

1.3 Reporting limits for most pesticides are 0.01-0.02 ug/L for aqueous samples, 20 ug/kg for soils and 0.05 mg/kg for wastes. Minimum reporting limits are highly matrix dependent and may not be achievable in all sample matrices. Refer to Attachment 23.3 (Table 2) for minimum reporting limits.

1.4 Instrumental calibration extends from the reporting limit to approximately 32 times the reporting limit. Quantitation may be extended by sample or extract dilution when necessary.

1.5 Pesticides analysis is restricted to use by or under the supervision of analysts experienced in gas chromatography, including chromatographic interpretation.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Revision 2, February, 2007*, Method 8081B, "Organochlorine Pesticides by Gas Chromatography"

2.2 *40 Code of Federal Regulations, most current edition, Part 136, Appendix A,* Method 608, "Organochlorine Pesticides and PCBs"

2.3 *Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume 1*, Method 608.2, "The Determination of Certain Organochlorine Pesticides in Municipal and Industrial Wastewater"

3.0 SUMMARY OF PROCEDURE

3.1 Samples are extracted using TriMatrix SOP GR-09-107 for water and SOP GR-09-108 for soil.

3.2 Extracts are injected onto the GC column equipped with an electron capture detector (ECD).

3.3 Quantitation is by comparing sample response to the response of standards in a six-point calibration. The extract volume injected is 1.0 µL.

4.0 PARAMETER OR COMPOUND LIST

UNCONTROLLED COPY

4.1 Target analytes are listed in Attachments 23.1 and 23.2 (Tables 1 and 1A).

4.2 Other analytes may be determined, provided each is analyzed in accordance with all procedural guidelines.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-03-101, *Semi-Volatile Laboratory Gas Chromatography Corrective Actions*, latest revision

5.2 TriMatrix SOP GR-09-105, *Waste Dilution Preparation Method for PCBs and Pesticides*, Method 3580A, latest revision

5.3 TriMatrix SOP GR-09-107, *Extraction of Organochlorine Pesticides and PCBs from Water*, Method 3510B, latest revision

5.4 TriMatrix SOP GR-09-108, *Extraction of PCBs/Pesticides from Soil, Sludge and Wastes*, Method 3550A, latest revision

5.5 TriMatrix SOP GR-09-111, *Florisil Column Cleanup*, Method 3620, latest revision

5.6 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.7 TriMatrix SOP GR-10-103, *Guidelines for Data Generation, Validation, Approval and Reporting*, latest revision

5.8 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

5.9 TriMatrix SOP GR-04-103, *Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Samples often contain materials that interfere with electron capture detectors. In addition, many samples contain substances that cause chromatographic interference on capillary columns. When necessary, Florisil clean-up is performed on extracts and corresponding quality control (QC).

6.2 When concentrated, solvents can also concentrate oxygenates and sulfur compounds, producing an ECD response. These compounds interfere with analyte peaks. It is imperative that pesticide grade solvents, ACS grade reagents and ultra-clean glassware be used at all times. For each solvent lot, an aliquot must be concentrated to analyze for such contamination and overall quality. Once a lot is approved for use, a six- month supply is sequestered and stored by the vender.

6.3 When extracted from samples or in standards exposed to water or methanol, kepone will produce a peak with a broad tail that elutes later than the calibration by up to one minute. This retention time shift will seriously affect the ability of the analyst to qualitatively identify this compound

UNCONTROLLED COPY

without being aware of the possibility. If there is any question about whether kepone is present, the analysis must be confirmed in accordance with TriMatrix SOP GR-04-103.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions on health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Pesticides can cause irritation upon contact and are suspected carcinogens. Take appropriate precautions when handling. A laboratory coat, disposable gloves and approved safety glasses must be worn while in the laboratory and when performing any laboratory operation associated with this procedure. Open and dispense concentrated standards only under a fume hood.

7.3 Solvents used in sample extraction and concentration are toxic. Avoid contact with skin, vapor inhalation and solvent ingestion. Always open solvent containers under a fume hood. A laboratory coat, disposable gloves and approved safety glasses must be worn when handling solvents.

7.4 All analyzed sample extracts and expired standards must be disposed of promptly and correctly.

7.5 If a pesticide or solvent spill occurs, contact the safety officer and/or area supervisor immediately then begin cleaning up the spill as outlined in the Safety Manual. Methylene chloride, pesticides, surrogates and spikes are highly toxic and suspected carcinogens. Wear all required protective clothing, disposable gloves and safety glasses to avoid contact. Prepare standards and samples under a fume hood.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Waters, wastewaters, and leachates are collected in unpreserved 1000 mL amber glass jars with PTFE-lined lids and stored at 4 ±2° C, until time of analysis. At least two 1000 mL aliquots of aqueous sample must be collected to meet quantitation limit requirements and allow for re-analysis when necessary. If matrix spikes are needed, collect two additional 1000 mL aliquots.

8.2 Wastes, sludges and soils are collected in inert bottles with no preservative added. At least 30 g of solid sample are needed to achieve default reporting limits. If insufficient sample is available the final extract volume can be reduced (minimum 1.0 mL) to achieve quantitation limits. Additional sample must be obtained for possible re-analysis and matrix spikes, as well as percent solids determinations.

8.3 Aqueous extractions must be performed within 7 days, and soil extractions within 14 days of sample collection. Extract analysis must be completed within 40 days of sample extraction.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Gas chromatograph equipped with at least one $_{63}$Ni electron capture detector (ECD) and flow valves capable of delivering very low flow rates (for example, 10 mL/minute) accurately. Refer to

UNCONTROLLED COPY

the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

9.1.1 Injector Temperature: 200° C

9.1.2 Detector Temperature: 350° C

9.1.3 Oven Temperature: Initial temperature at 120° C then ramp to 160° C at 20° C/min then ramp to 300° C at 7° C/min. Hold for 4 minutes at 300° C

9.2 A personal computer capable of storing and retrieving chromatographic data. Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer specifications associated with the analytical instrument.

9.3 Columns:

9.3.1 Column A: RTX CLP (or equivalent), 0.53 mm ID, 0.50 um film thickness, 30 m length

9.3.2 Column B: RTX CLP2 (or equivalent), 0.53 mm ID, 0.42 um film thickness, 30 m length

9.4 Various size microsyringes

9.5 Autosampler vials with PTFE-faced silicone septa, 1.5 mL

9.6 Volumetric flasks

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Septa replacement:

10.1.1 Direct inject gas chromatographs are equipped with silicone septa (Supelco Thermogreen 10 mm or equivalent) which eventually core and leak. These must be replaced to avoid contaminating the inlet sleeve with rubber particles and to minimize bleed. Replace weekly or after approximately 100 injections.

10.2 Inlet Sleeve Cleaning and/or Replacement

10.2.1 Inlet sleeves can become a source of contamination, flow restriction and pesticide breakdown as they accumulate sample debris and septum particles over time. When an Endrin/DDT breakdown check standard indicates 15% or higher breakdown, the sleeve must be cleaned or replaced.

10.3 Column Clipping/Replacement

10.3.1 In addition to sleeve replacement and when conditions merit, about 6 inches can be clipped off the front of the guard column. When a guard column becomes too short to clip, it must be replaced. If resolution is lost and/or breakdown is high after a guard column is replaced, the analytical column must also be clipped/replaced.

UNCONTROLLED COPY

10.4 ECD Wipe Test and $_{63}$Ni Foil Cleaning/Replacement

10.4.1 Electron capture detectors may begin to bleed radioactive particles over time. Wipe tests must be conducted every six months to ensure that bleed is within acceptable levels.

10.4.2 Sample components and constant use at high temperature depletes the $_{63}$Ni foil. This raises baseline noise and decreases the linear range of the detector. When this happens, the detector must be sent out for cleaning and/or re-foiling. **NEVER at any time may an unlicensed person tamper with the $_{63}$Ni foil or any internal ECD part. This must be done by a licensed professional, trained in working with radioactive materials.**

11.0 CHEMICALS AND REAGENTS

11.1 Pesticide standards, purchased as certified standards in ampules from Ultra-Scientific, Restek, Absolute or other certified manufacturers.

11.2 Hexane, Pesticide Grade or equivalent

11.3 Helium carrier gas, ultra high purity, 2-3 mL/minute

11.4 Nitrogen makeup gas, filtered for oxygen, hydrocarbons and moisture, 40-60 mL/minute

12.0 STANDARDS PREPARATION

12.1 Prepare stock standards as follows:

12.1.1 Stock standards are purchased as 1000 mg/L certified solutions. The primary standard for the normal pesticides list is purchased from Ultra Scientific at 1000 ug/mL (Catalog PPM-808C).

12.1.2 The second-source standard is purchased from Chem Service at 1000 ug/mL (Catalog OCP508-1JM).

12.1.3 Additional pesticides listed in Table 1A are purchased as individual standards and are combined as necessary for intermediate and calibration standards. Multi-component standards of technical chlordane and toxaphene are not combined.

12.1.4 Transfer any unused and opened stock solution to a 1.5 mL amber screw-cap vial after opening the original ampule. Store at 4 ±2° C with zero headspace and protect from light.

12.1.5 All stock standards must be replaced at one year from the date opened, the earliest individual compound expiration date or sooner if comparison with routine quality control indicates a problem.

UNCONTROLLED COPY

12.1.6 Purchased standards may only be used if certified by the manufacturer or an independent source as traceable to NIST.

12.3 If unable to purchase as a certified solution, prepare non-standard analytes gravimetrically from the neat material as follows:

12.3.1 To prepare a standard at approximately 1000 mg/L, weigh approximately 0.1 g (100 mg) of compound into a 100 mL volumetric. Add hexane (some toluene may be necessary to dissolve certain pesticides) and shake until dissolved. Calculate concentration as follows:

$$\frac{\text{mg compound}}{0.100\,\text{L solvent}} = mg/L\,(\text{or ug/mL})$$

For Example:

$$\frac{101\,\text{mg Mirex}}{0.100\,\text{L Hexane}} = 1010\,mg/L\,\text{Mirex}$$

12.4 Prepare intermediate standard from the stock solution as follows:

12.4.1 Prepare in hexane.

12.4.2 Prepare at a concentration that is the highest calibration point in the curve (0.16 mg/L).

Initial Concentration (mg/L)	Standard Volume needed (µL)	Final Volume (mL)	Final Concentration (mg/L)
1000	8.00	50	0.16

12.4.3 Expiration is six months from the date prepared and/or no later than the stock standard expiration date or when routine quality control indicates a problem. Whichever is first.

12.5 Prepare calibration standards as follows:

12.5.1 Prepare in hexane.

12.5.2 These dilutions are used for the initial calibration, second-source verification and continuing calibration verification standards.

12.5.3 Serial dilution of the intermediate standard is based on adding a volume of standard to a volume of hexane:

UNCONTROLLED COPY

Initial Concentration (mg/L)	Standard Volume needed (µL)	Hexane Volume added to Standard Volume (µL)	Final Volume (mL)	Final Concentration (mg/L)
0.16	500	500	1	0.08
0.08	500	500	1	0.04
0.04	500	500	1	0.02
0.02	500	500	1	0.01
0.01	500	500	1	0.005

12.6 Label all standards with the following information:

12.6.1 Concentration with units

12.6.2 Parameter

12.6.3 The standards log identification as it appears in the laboratory information management system (Element™)

12.6.3 Expiration date

12.7 All standards and standard dilutions must be recorded in Element™.

13.0 SAMPLE PREPARATION

13.1 For detailed sample extraction and clean-up, refer to the appropriate TriMatrix SOP listed in Section 5.0.

14.0 CALIBRATION PROCEDURES

14.1 Initial Calibration of single-component pesticides:

14.1.1 Following the daily hexane blank, inject a 0.05/0.10 mg/L endrin/DDT breakdown standard. Percent breakdown for endrin and DDT must be less than 15%. These criteria must be met before subsequent injections are made. Repeat the degradation check every 12 operational hours. Refer to Section 10.0 for maintenance instructions if a breakdown check fails. Calculate percent breakdown for endrin and DDT where Hx = peak height of the designated compound:

$$\% \text{ Endrin Breakdown} = \frac{H_{EA} + H_{EK}}{H_{EA} + H_{EK} + H_E} \times 100$$

Where:
H_{EA} = Endrin aldehyde peak height

UNCONTROLLED COPY

H_{EK} = Endrin ketone peak height
H_E = Endrin peak height

$$\% \text{ DDT Breakdown} = \frac{H_{DDE} + H_{DDD}}{H_{DDE} + H_{DDD} + H_{DDT}} \times 100$$

Where:
H_{DDE} = DDE peak height
H_{DDD} = DDD peak height
H_{DDT} = DDT peak height

14.1.2 After the breakdown check, inject the calibration standards (0.005, 0.010, 0.020, 0.040, 0.080, 0.160 mg/L). Refer to Attachment 23.6 (Figure 2) for an example DDT calibration curve.

14.1.3 Calculate the calibration factors (CF) for each analyte:

$$CF = \frac{\text{Peak Height}}{\text{Analyte Concentrat ion in ng/uL}}$$

14.1.4 To evaluate linearity through the origin, calculate percent relative standard deviation (%RSD) for each analyte:

$$\% RSD = \frac{\text{Standard Deviation}}{\text{Average CF}} \times 100$$

14.1.5 If %RSD is less than or equal to 20% using all six standards, the calibration curve is assumed linear through the origin and the average calibration factor may be used for quantitation.

14.1.6 If %RSD is greater than 20%, a regression curve must be used. Depending on the coefficient of determination, least squares regression (first order) or polynomial (second order) regression may be used. Linearity through the origin cannot be assumed and the regression must not be forced through the origin.

14.1.6.1 Linear regression (first order) is a plot of peak height versus standard concentration and is applicable when data exhibits linearity that is not through the origin. This curve is evaluated using the Turbochrom coefficient of determination (r^2). The minimum acceptable coefficient of determination for a linear regression calibration is 0.990. The linear regression equation is as follows:

$$x = \frac{(y - b)}{m}$$

where:
x = Analyte concentration in the extract, in mg/L

UNCONTROLLED COPY

y = Analyte peak height in the extract
m = The regression line slope
b = The regression line y-intercept

14.1.6.2 Non-linear regression (second order) is used when linear regression has a coefficient of determination greater than 0.990. Evaluate the second order fit using coefficient of determination (r^2) with the same limit of 0.990. The equation for second order regression is as follows but must be solved by the instrument or by the function "linest" (=linest(y-values,x-values^{1,2},,true)) in an Excel spreadsheet with calculates coefficients a, b and c:

$$y = ax^2 + bx + c$$

where:
x = Concentration of pesticide in the extract
y = Peak height of pesticide in the extract
a = The coefficient corresponding to the square of the x-value
b = The coefficient corresponding to the x-value
c = A constant

14.1.7 A regression calibration may be used when %RSD is less than 20% but the regression plot does not go close enough to the origin to provide a sufficiently accurate quantitation.

Note: Do not use non-linear regression to compensate for detector saturation in place of instrument maintenance. Non-linear regression must be employed only for analytes and/or instruments previously shown to not exhibit linear calibration at optimum instrument operating conditions. Quantitation by average CF and/or linear regression must not be changed to non-linear calibration to compensate for instrument maintenance requirements.

14.1.8 Inject the second-source calibration verification (SCV) standard immediately following the initial calibration. All analytes must have recoveries between 75-125% for the calibration to be considered valid. If these criteria are not met, re-analyze the SCV a second time. If it still does not pass, take corrective action to locate and solve the problem up to and including preparing fresh standards from fresh stock. Then run an acceptable initial calibration.

14.1.9 Analysis may not begin without an acceptable initial calibration.

14.1.10 Refer to Attachment 23.6 (Figure 2) for an example calibration curve for DDT.

14.1.11 When calibrating for samples being reported under EPA 608 or 608.2 (methoxychlor), there are different requirements for calibration, which include the following:

14.1.11.1 Initial calibration may consist of a minimum of three concentration points instead of six.

UNCONTROLLED COPY

Note: This minimum is superseded by any client or agency requesting more calibration points for quantitation.

14.1.11.2 Percent relative standard deviation (%RSD) of an average calibration factor must be less than 10% instead of less than 20%, to be acceptable for quantitation.

14.1.11.3 A linear regression curve must be constructed if the calibration factor %RSD is greater than or equal to 10%. The coefficient of determination (r^2) must be at least 0.990.

14.1.11.4 Non-linear regression is not allowed with only 3 calibration points.

14.2 Prepare and use the initial calibration of chlordane and toxaphene as follows:

14.2.1 Chlordane and toxaphene are multicomponent pesticides which require additional calibration instructions.

14.2.2 Run a 0.1 mg/L single-point standard for chlordane as a pattern-recognition standard.

14.2.3 Run a 1.0 mg/L single-point toxaphene standard as a pattern-recognition standard. The six point calibration does not need done if there is no pattern match in any extract being analyzed unless otherwise specified.

14.2.4 If chlordane or toxaphene is detected, calibrate using a 6-point calibration (3-point for method 608) as with single-component pesticides. Re-run the detected analyte and quantitate using the 6-point initial calibration.

14.2.5 Quantitate by using the sum of five characteristic peak heights:

$$CF = \frac{\text{Sum of Characteristic Peak Height}}{\text{Concentration of Multi-Component Analyte}}$$

14.3 Calibration Verification

14.3.1 On non-curve days, run a calibration verification standard containing all analytes of interest (including chlordane and toxaphene) following the daily blank or blank spike (BS), and the degradation check standard.

14.3.2 Quantitate in a manner consistent with how the initial calibration was approved (average CF or regression):

$$Concentration(mg/L) = \frac{\text{Height Counts of the Analyte}}{\text{Average CF}}$$

14.3.1.1 For average CF, verify by percent difference between the average and continuing factors using the following equation:

UNCONTROLLED COPY

$$Percent\ Difference = \frac{CF_v - CF_m}{CF_m} \times 100$$

where:

CF_v = Response factor from verification standard
CF_m = Average calibration factor from initial calibration

14.3.1.2 For regression, verify by calculating percent drift between the actual and quantitated standard using the following equation:

$$\text{Percent Drift} = \frac{(R_2 - R_1)}{R_1}$$

where:

R_1 = Actual value
R_2 = Quantitated value

14.3.2 Percent difference or percent drift must be within ±20% of the actual value for samples analyzed by method 8081B. Percent difference or percent drift must be within ±15% of the actual value for samples analyzed by method 608. Percent difference or percent drift must be within ±10% of the actual value for samples analyzed by method 608.2 (methoxychlor).

15.0 ANALYTICAL PROCEDURE

15.1 The analysis sequence begins with a hexane run (BLK) followed by the degradation check, then the initial continuing calibration verification (CCV) which is a same-source calibration standard.

15.2 After every 10 sample injections, analyze a continuing calibration verification (CCV) standard at the same concentration as the initial CCV. Each analyte must be within criteria specified in section 14.3.2 on both columns or all associated samples need reanalyzed with an in-control CCV except for the following exception:

15.2.1 If CCV response is outside the upper acceptance limit and analyte is not detected in the associated samples then those samples do not need to be reanalyzed. The CCV outside the upper acceptance limit still demonstrates that analyte will be detected if present.

15.2.2 However, reanalyze all associated samples with any analyte that is detected even for this exception.

15.3 Identify pesticides by comparing peak retention times in the chromatogram with standard peak retention times. For a positive identification, the analyte retention time must elute within the calculated retention time window.

UNCONTROLLED COPY

15.4 For each target analyte, a retention time window must be calculated:

15.4.1 Make three injections over a minimum of a 72-hour period and record the retention times of each. A retention time spreadsheet is located on the laboratory intranet library.

15.4.2 Calculate standard deviation of the average retention time. For toxaphene and chlordane, choose the major peak.

15.4.3 Calculate the retention time limit by multiplying the standard deviation by three. The retention time window is ±3 times the standard deviation.

15.4.4 All reportable analyte quantitations must elute within the retention time of the first calibration verification of the day, plus/minus the retention time limit.

15.4.5 All injections run after the first calibration verification of the day must pass retention time window criteria. Any sample extract injected after a retention time window failure must be re-injected.

15.4.6 For toxaphene and chlordane, use retention time windows for quantitation but rely primarily on pattern recognition for qualitative identification.

15.4.7 Re-run the retention time window study when a new column is installed, significant changes in the instrument occur or when operating conditions change.

15.5 Example chromatograms showing elution orders on RTX CLP and RTX CLP2 columns are provided in Attachment 23.7 (Figure 3), Attachment 23.8 (Figure 4) and Attachment 23.9 (Figure 5).

15.6 All analyses must be injected within 24 hours from the first injection of the day which is the hexane blank or blank spike. Analyses injected outside the 24-hour window must be re-injected. The degradation check standard must be injected at a minimum of every 12 hours to verify system inertness.

15.7 All targeted analyte identifications must be confirmed by running on the secondary column or by gas chromatography/mass spectrometry (GCMS).

15.7.1 When confirmation is made using a second column, the secondary analysis must meet all QC criteria described for the primary column, including calibration and retention times. Evaluate the agreement between results after identification has been confirmed by relative percent difference.

15.7.2 Calculate relative percent difference (RPD) using the formula below where R_1 and R_2 are the two results. The vertical bars in the numerator indicate the absolute value of the difference. Therefore, RPD is always a positive value.

.

$$RPD = \frac{|R_1 - R_2|}{R_1 + R_2} 200$$

UNCONTROLLED COPY

15.7.3 If one result is significantly higher (if RPD is greater than 40%), check to see if an obviously overlapping peak is causing one erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. If no anomalies are noted, review the chromatographic conditions.

15.7.4 If there is no evidence of chromatographic problems such as overlapping peaks, report the lower result (unless otherwise specified) with narration as to the disparity between column results.

15.7.5 If RPD is less than 40%, report from the primary column.

15.8 Check reportable results for being a false positive due to poor integration or peak integrity (more blob than peak) on both columns. Manual integration must only be performed in strict adherence to TriMatrix SOP GR-10-115.

15.9 If peak response exceeds the calibration range, prepare a dilution of the extract and re-inject. Dilute only enough to bring peak responses exceeding the calibration into the middle of the calibration range.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Quantitate using average CF or regression as discussed in Section 14.0.

16.2 The instrument software applies the appropriate equation to calculate ng/uL (mg/L) of extract concentration:

$$ng/uL(AverageCF) = \frac{PeakHeight(s)}{CF}$$

$$ng/uL(Linear) = \frac{v-b}{m}$$

a, b, c coefficients (nonlinear/Excel) = linest(y-values,x-values^{1,2},,true)

16.3 Next, calculate sample concentration:

For water samples:

$$sample\ concentration\ (ug/L) = \frac{(ng/uL)(V_e)(DF)}{V_w}$$

For soil samples:

UNCONTROLLED COPY

$$sample\ concentration\ (mg/kg) = \frac{(ng/uL)(V_e)(DF)(1000)}{V_s(PS)}$$

where:

V_e = Total extract volume (µL)
DF = Post-extraction dilution factor (µL + µL)/(total µL)
V_w = Initial sample volume (mL)
V_s = Initial soil sample mass (g)
PS = Soil solids fraction (use 0.98, not 98%)

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for data quality and for filling in all documentation and paperwork correctly. It is important to document analysis by correctly filling in, handing in and archiving all paperwork correctly.

17.2 Laboratory Information Management System (Element™) data transfer must be in accordance with TriMatrix SOP GR-10-123. If internal chain-of-custody is required, it is very important that a CoC form be filled in correctly and completely.

17.3 All run, maintenance, CD archival and standard logs must be filled in completely and correctly. Corrections on hardcopy must be made with a lineout (not a writeover) and must be initialed and dated. Blank lines in run logbooks must be Z'd out. Refer to Attachment 21.12 for a run logbook example.

17.4 All calibration run hardcopy must be archived in the correct storage box.

17.5 All other analysis documentation must be archived appropriately for ease in retrieval.

18.0 QUALITY ASSURANCE

18.1 In addition to quality control in previous sections of this procedure, the following QC must be included:

18.2 Blanks

18.2.1 Before calibration or the first calibration verification of the day (CCV) is run, a solvent blank (BLK) is required to show the system is free from interferences and contamination is at or below the reporting limit. The BLK is hexane, spiked with surrogates. At a minimum, the solvent blank must be run at the beginning of each 24-hour shift. The solvent blank will be injected more frequently if contamination is suspected from a high level extract or if laboratory contamination is in question.

18.2.2 When contamination is found above any analyte reporting limit, re-inject the blank. If contamination is still observed, determine the source of interference and correct before proceeding with calibration or the initial CCV. If the contamination source cannot be

UNCONTROLLED COPY

located, notify the laboratory supervisor. Do not analyze sample extracts on a contaminated system.

18.2.3 An extraction blank (BLK) is organic-free laboratory reagent water for water samples or sodium sulfate for soil samples:

18.2.3.1 The extraction blank is carried through every step in the extraction, including extract cleanup (if any).

18.2.3.2 At least one extraction blank must be extracted with each extraction batch.

18.2.3.3 If there are reportable results in the extraction blank but all associated sample extracts are non-detect, their data may be used but must be narrated.

18.2.3.4 If there are reportable results in sample extracts, re-extraction of all associated samples and extraction quality control must be performed when sample volume permits. If not, consult the area supervisor and/or the project chemist.

18.3 Surrogates

18.3.1 All solvent blanks and extracts must be spiked with decachlorobiphenyl and tetrachloro-m-xylene surrogates before injection.

18.3.2 Until 20 samples of a given matrix are analyzed, the recovery window will be 50-150%. Once 20 samples of a given matrix have been analyzed, laboratory recovery limits must be generated and used. Surrogate recovery windows must be updated annually, by matrix.

18.3.2.1 Calculate surrogate recovery for each extract injected.

18.3.2.2 If recovery is not within acceptable limits, consult TriMatrix SOP GR-03-101 to determine when and how data is qualified.

18.3.3 Upper and lower control limits for each surrogate are generated and maintained in Element™ as follows:

Upper Control Limit (UCL) = p + 3s

Lower Control Limit (LCL) = p - 3s

where:
p = Average percent recovery
s = Standard deviation of the average percent recovery

18.3.4 Use two standard deviations as surrogate control limits when three standard deviations give a negative lower control limit.

UNCONTROLLED COPY

18.4 Analyze matrix spikes and blank spikes as follows:

18.4.1 To assess extraction efficiency, extract a matrix spike (MS)/matrix spike duplicate (MSD) and a blank spike (BS) at least once every 20 samples extracted, per matrix. If one to ten samples are analyzed in a month, at least one matrix spike and matrix spike duplicate is required. A blank spike must be extracted daily.

18.4.2 Until 20 matrix spike/matrix spike duplicates and blank spikes have been analyzed, matrix spike/matrix spike recovery and duplication, and blank spike recovery must be monitored against a default window of 50-150% and a maximum limit of 20% as RPD.

18.4.3 Statistical recovery control limits are calculated and maintained in Element™. Analyte spike recovery is based on the following calculation:

$$\%\ recovery = \frac{(Aspk - Asmp)}{SPK} \times 100$$

where:
A_{spk} = Actual concentration found in the spiked sample in ug/L
A_{smp} = Concentration found in non-spiked sample in ug/L

$$SPK = \left(\frac{concentration\ of\ spiked\ standard\ in\ ug/L \times ml\ spiked}{initial\ sample\ volume\ in\ L} \right)$$

18.4.4 The statistical duplication control limit is calculated and maintained in Element™. Analyte spike duplication is based on the following calculation:

$$\%RPD = \frac{\left|(SPK_1 - SPK_2)\right|}{\left[\frac{(SPK1 + SPK2)}{2}\right]} \times 100$$

where:
SPK_1 = ug/L found in matrix spike (MS)
SPK_2 = ug/L found in matrix spike duplicate (MSD)

18.4.5 Control limits must be updated at least annually.

18.4.6 If recovery or precision is not within acceptance limits, refer to TriMatrix SOP GR-03-101 for corrective action and whether data must be narrated as qualified.

18.4.7 If the blank spike is out-of-control, the problem must be immediately identified and corrected before further samples for that analyte can be run or extracted.

18.4.7.1 The purpose of the blank spike is to verify that out-of-control recoveries in the MS/MSD are the result of matrix interference rather than extraction or system error.

UNCONTROLLED COPY

18.4.7.2 Failure of any part of a blank spike requires corrective action, depending on the failed parameter.

18.4.7.3 A non-conformance report must be completed and every effort must be made to determine the root cause of the failure (examples: mis-spiked, mis-extracted).

18.5 Field duplicates are specifically required for methoxychlor analysis, by method 608.2

18.5.1 A field duplicate analysis must be performed every 10 samples.

18.5.2 Samples with reportable methoxychlor within the extraction batch must be spiked if possible. Otherwise, choose a sample at random.

18.5.3 Calculate relative range (RR_i) for the field duplicate as follows:

$RR_i\,(\%) = (100*R_i)/[(X_1 + X_2)/2]$

Where:
R_i = Absolute difference between the duplicate measurements X_1 and X_2 in ug/L
X_i = Average concentration found $[(X_1 + X_2)/2]$

18.5.4 Until enough data is available to calculate statistical control limits, acceptability must be based on a maximum of 20% RR_i for samples having reportable methoxychlor concentrations.

18.6 Corrective action for out-of-control data must be performed in accordance with TriMatrix SOP GR-03-101.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is also required on each instrument used to demonstrate the instrument's ability to generate acceptable accuracy and precision.

19.1.1 The initial demonstration of capability (IDC) may be accomplished as follows:

19.1.1.1 Spike four 1000 mL aliquots of water or four 30 g aliquots of sodium sulfate so the resulting extract concentration is in the lower half of the calibration.

19.1.1.2 Process the four spiked aliquots through every step outlined in the extraction, clean-up and analysis procedures.

19.1.1.3 Input the four results into the IDC spreadsheet located on the laboratory intranet library in ug/L or mg/kg.

UNCONTROLLED COPY

19.1.1.4 The spreadsheet will calculate average percent recovery and standard deviation.

19.1.1.5 Average percent recovery must be within the blank spike control limits listed in Element™.

19.1.1.6 Relative standard deviation must be less than or equal to 20% as RSD.

19.1.1.7 If either criterion is not met, locate and correct the source of the problem and repeat the study successfully.

19.1.1.8 Repeated failure will confirm a general problem with the procedure and/or techniques used. If this occurs, locate and correct the source of the problem, revise the procedure and/or techniques used then repeat the study successfully.

19.1.1.9 Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed.

19.1.1.10 Give a copy of all IDC study attempts to the Quality Assurance Department.

19.1.2 A continuing demonstration of capability (CDC) is required annually by one of the following approaches:

19.1.2.1 By repeating the IDC study.

19.1.2.2 By inputting 4 consecutive blank spike results obtained during the course of routine sample extraction/analysis into the IDC spreadsheet.

19.1.2.3 By extracting/analyzing a successful blind performance evaluation sample during the course of routine sample extraction/analysis.

19.1.2.4 By inputting the last four results from a method detection limit study into the IDC spreadsheet if exclusively analyzed by the analyst.

19.1.2.5 Give a copy of all CDC attempts to the Quality Assurance Department.

19.2 A Method Detection Limit (MDL) study must be performed annually in accordance with TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material. Refer to TriMatrix SOP GR-15-102.

20.3 Conserve the use of chemicals where applicable.

UNCONTROLLED COPY

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Revision 2, February, 2007*, Method 8081B, "Organochlorine Pesticides by Gas Chromatography"

22.2 *40 Code of Federal Regulations, most current edition, Part 136, Appendix A,* Method 608, "Organochlorine Pesticides and PCBs"

22.3 *Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume 1*, Method 608.2, "The Determination of Certain Organochlorine Pesticides in Municipal and Industrial Wastewater"

23.0 ATTACHMENTS

23.1 Table 1 – Targets and Surrogates, Retention Times and CAS Numbers

23.2 Table 1A – Additional Targets, Retention Times and CAS Numbers

23.3 Table 2 – Minimum Reporting Limits

23.4 Table 3 – Method Detection Limit Study Example

23.5 Figure 1 – Analytical Standards Report Example

23.6 Figure 2 – DDT Calibration Curve Example

23.7 Figure 3 – Pesticide Chromatogram on RTX CLP Column

23.8 Figure 4 – Pesticide Chromatogram on RTX CLP2 Column

23.9 Figure 5 – Chlordane Multi-Component Pesticide Chromatogram

23.10 Figure 6 – Data Review Report Example

UNCONTROLLED COPY

23.11 Figure 7 – Preparation Batch Detail Report Example

23.12 Figure 10 – Instrument Run Log Example

UNCONTROLLED COPY

Table 1
Targets and Surrogates, Retention Times and CAS Numbers

Analyte	RTX CLP	RTX CLP2	CAS #
Aldrin	12.37	13.77	309-00-2
a-BHC	10.17	11.32	319-84-6
b-BHC	10.98	12.25	319-95-7
d-BHC	11.34	12.90	319-86-8
g-BHC, (Lindane)	10.82	12.10	58-89-9
4,4'-DDD	15.26	16.95	72-54-8
4,4'-DDE	14.15	15.83	72-55-9
4,4'-DDT	15.78	17.57	50-29-3
Dieldrin	14.74	16.24	60-57-1
Endosulfan I	14.29	15.71	959-98-8
Endosulfan II	15.59	17.24	33212-65-9
Endosulfan sulfate	17.17	18.41	1031-07-8
Endrin	15.18	16.85	72-20-8
Endrin aldehyde	16.37	17.87	7421-93-4
Heptachlor	11.78	13.08	76-44-8
Heptachlor epoxide	13.54	14.91	1024-57-3
Methoxychlor	16.66	18.83	72-43-5
a-Chlordane	14.02	15.57	5103-71-9
g-Chlordane	13.77	15.28	5103-74-2
Endrin ketone	17.70	19.47	53494-70-5
Decachlorobiphenyl (surr)	19.52	22.07	2051-24-3
Tetrachloro-m-xylene (surr)	9.01	9.93	877-09-8

**Multi-response compound

Table 1A
Additional Targets, Retention Times and CAS Numbers

Analyte	RTX CLP	RTX CLP2	CAS #
Diallate	9.79	10.74	2303-16-4
Isodrin	13.03	12.88	465-73-6
Kepone	15.32	15.34	143-50-0
Mirex	15.86	17.63	72-43-5
Chlorobenzilate	15.28	15.02	510-15-6
2,4-DDT	16.68	19.02	-
2,4-DDD	-	-	-
2,4-DDE	-	-	-
2,2'-4,4'-5,5'-Hexabromobiphenyl (BP-6)	23.46	27.84	-
Hexabromobenzene (HBB)	18.01	21.74	-
Tris-(2,3-dibromoprpryl) phosphate	**	**	-
Chlordane, Technical Grade	**	**	57-74-9
Toxaphene	**	**	8001-35-2

**Multi-response compound

UNCONTROLLED COPY

Table 2
Minimum Reporting Limits

Compound	Water (ug/L)	Soil (mg/kg)
Aldrin	0.010	0.0017
a-BHC	0.010	0.0017
b-BHC	0.010	0.0017
d-BHC	0.010	0.0017
g-BHC, (Lindane)	0.010	0.0017
4,4'-DDD	0.010	0.0017
4,4'-DDE	0.010	0.0017
4,4'-DDT	0.010	0.0017
Dieldrin	0.010	0.0017
Endosulfan I	0.010	0.0017
Endosulfan II	0.010	0.0017
Endosulfan sulfate	0.010	0.0017
Endrin	0.010	0.0017
Endrin aldehyde	0.010	0.0017
Heptachlor	0.010	0.0017
Heptachlor epoxide	0.010	0.0017
Methoxychlor	0.010	0.0017
Toxaphene	0.050	0.0170
a-Chlordane	0.010	0.0017
g-Chlordane	0.010	0.0017
Endrin ketone	0.010	0.0017
Diallate	0.020	0.0033
Isodrin	0.010	0.0017
Kepone	0.010	0.0017
Mirex	0.010	0.0017
Chlorobenzilate	0.010	0.0017
Chlordane, Technical Grade	0.025	0.0083

UNCONTROLLED COPY

Laboratories, Inc.

SEMI-VOLATILE/VOLATILE/METALS LABORATORY
INSTRUMENT NUMBER 158A 2004 WATER
METHOD DETECTION LIMIT STUDY

nd	Reference Citation	Date Analyzed	Amount Spiked	Units	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Rep. #6	Rep. #7	Average Amount Found	Average % Recovery	Standard Deviation	MDL
	608/8081A	1/10/2004	0.008	ug/L	0.007	0.007	0.006	0.007	0.004	0.007	0.008	0.007	85%	0.0013	0.0040
	608/8081A	1/10/2004	0.008	ug/L	0.007	0.008	0.007	0.007	0.004	0.007	0.008	0.007	88%	0.0012	0.0038
	608/8081A	3/15/2004	0.008	ug/L	0.007	0.007	0.006	0.007	0.005	0.001	0.006	0.005	69%	0.0021	0.0067
	608/8081A	1/10/2004	0.008	ug/L	0.007	0.007	0.007	0.007	0.004	0.007	0.007	0.007	82%	0.0011	0.0036
	608/8081A	1/10/2004	0.008	ug/L	0.008	0.009	0.009	0.010	0.006	0.009	0.010	0.009	108%	0.0015	0.0047
	608/8081A	3/15/2004	0.008	ug/L	0.005	0.005	0.004	0.004	0.004	0.003	0.004	0.004	53%	0.0006	0.0019
	608/8081A	1/10/2004	0.008	ug/L	0.009	0.008	0.008	0.008	0.005	0.008	0.009	0.008	99%	0.0014	0.0043
	608/8081A	1/10/2004	0.008	ug/L	0.011	0.008	0.008	0.008	0.006	0.010	0.009	0.008	106%	0.0018	0.0057
	608/8081A	1/10/2004	0.008	ug/L	0.007	0.008	0.007	0.007	0.005	0.007	0.008	0.007	85%	0.0011	0.0035
	608/8081A	3/15/2004	0.008	ug/L	0.010	0.010	0.009	0.010	0.009	0.005	0.008	0.009	106%	0.0019	0.0058
	608/8081A	3/15/2004	0.008	ug/L	0.010	0.010	0.009	0.010	0.009	0.005	0.008	0.009	106%	0.0019	0.0058
	608/8081A	1/10/2004	0.008	ug/L	0.009	0.009	0.008	0.008	0.005	0.008	0.009	0.008	99%	0.0014	0.0044
	608/8081A	1/10/2004	0.008	ug/L	0.010	0.010	0.010	0.010	0.007	0.012	0.013	0.010	130%	0.0017	0.0055
	608/8081A	3/15/2004	0.008	ug/L	0.010	0.010	0.009	0.010	0.009	0.006	0.009	0.009	115%	0.0015	0.0047
	608/8081A	1/10/2004	0.008	ug/L	0.005	0.004	0.004	0.005	0.003	0.005	0.006	0.004	55%	0.0009	0.0028
	608/8081A	1/10/2004	0.008	ug/L	0.009	0.009	0.008	0.008	0.005	0.009	0.009	0.008	102%	0.0014	0.0044
	608/8081A	3/17/2004	0.008	ug/L	0.011	0.009	0.009	0.012	0.008	0.004	0.009	0.009	109%	0.0025	0.0078
	608.2/8081A	1/10/2004	0.008	ug/L	0.013	0.013	0.011	0.012	0.008	0.014	0.012	0.012	146%	0.0020	0.0063
	608/8081A	1/10/2004	0.008	ug/L	0.009	0.009	0.008	0.008	0.005	0.009	0.009	0.008	101%	0.0014	0.0045
	608/8081A	1/10/2004	0.008	ug/L	0.011	0.011	0.010	0.010	0.006	0.011	0.011	0.010	126%	0.0018	0.0056

1-29-10
QA Officer

Approved By: JWB 2-10-10
Area Supervisor

CONFIDENTIAL

Figure 1
Analytical Standards Report Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
7090175

Description:	pest L1	Expires:	Jan-02-08
Standard Type:	Calibration Stan	Prepared:	Sep-06-07
Solvent:	Solvent Lot #	Prepared By:	Daniel J. Mierendorf
Final Volume (mls):	2	Department:	Semivolatiles GC
Vials:	1	Last Edit:	Sep-06-07 16:42 by DJM

Analyte	CAS Number	Concentration	Units
alpha-Chlordane	5103-71-9	0.005	ug/mL
4,4'-DDD	72-54-8	0.005	ug/mL
Dieldrin [2C]	60-57-1	0.005	ug/mL
Dieldrin	60-57-1	0.005	ug/mL
delta-BHC [2C]	319-86-8	0.005	ug/mL
delta-BHC	319-86-8	0.005	ug/mL
Decachlorobiphenyl [2C]	2051-24-3	0.005	ug/mL
Decachlorobiphenyl	2051-24-3	0.005	ug/mL
beta-BHC [2C]	319-85-7	0.005	ug/mL
Endosulfan I [2C]	959-98-8	0.005	ug/mL
alpha-Chlordane [2C]	5103-71-9	0.005	ug/mL
Endosulfan II	33213-65-9	0.005	ug/mL
alpha-BHC [2C]	319-84-6	0.005	ug/mL
alpha-BHC	319-84-6	0.005	ug/mL
Aldrin [2C]	309-00-2	0.005	ug/mL
Aldrin	309-00-2	0.005	ug/mL
4,4'-DDT [2C]	50-29-3	0.005	ug/mL
4,4'-DDT	50-29-3	0.005	ug/mL
4,4'-DDE [2C]	72-55-9	0.005	ug/mL
4,4'-DDE	72-55-9	0.005	ug/mL
4,4'-DDD [2C]	72-54-8	0.005	ug/mL
beta-BHC	319-85-7	0.005	ug/mL
gamma-BHC (Lindane)	58-89-9	0.005	ug/mL
Tetrachloro-m-xylene	877-09-8	0.005	ug/mL
Methoxychlor [2C]	72-43-5	0.005	ug/mL
Methoxychlor	72-43-5	0.005	ug/mL
Heptachlor Epoxide [2C]	1024-57-3	0.005	ug/mL
Heptachlor Epoxide	1024-57-3	0.005	ug/mL
Heptachlor [2C]	76-44-8	0.005	ug/mL
Heptachlor	76-44-8	0.005	ug/mL

Reviewed By _____ Date _____

Page 1 of 2

Analytical Standard Record
TriMatrix Laboratories, Inc.
7090175

gamma-Chlordane [2C]	5103-74-2	0.005	ug/mL
Endosulfan I	959-98-8	0.005	ug/mL
gamma-BHC (Lindane) [2C]	58-89-9	0.005	ug/mL
Tetrachloro-m-xylene [2C]	877-09-8	0.005	ug/mL
Endrin Ketone [2C]	53494-70-5	0.005	ug/mL
Endrin Ketone	53494-70-5	0.005	ug/mL
Endrin Aldehyde [2C]	7421-93-4	0.005	ug/mL
Endrin Aldehyde	7421-93-4	0.005	ug/mL
Endrin [2C]	72-20-8	0.005	ug/mL
Endrin	72-20-8	0.005	ug/mL
Endosulfan Sulfate [2C]	1031-07-8	0.005	ug/mL
Endosulfan Sulfate	1031-07-8	0.005	ug/mL
Endosulfan II [2C]	33213-65-9	0.005	ug/mL
gamma-Chlordane	5103-74-2	0.005	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
7090174	pest L2	Sep-06-07	Daniel J. Mierendo	Jan-02-08	Sep-06-07 16:42 by DJM	1

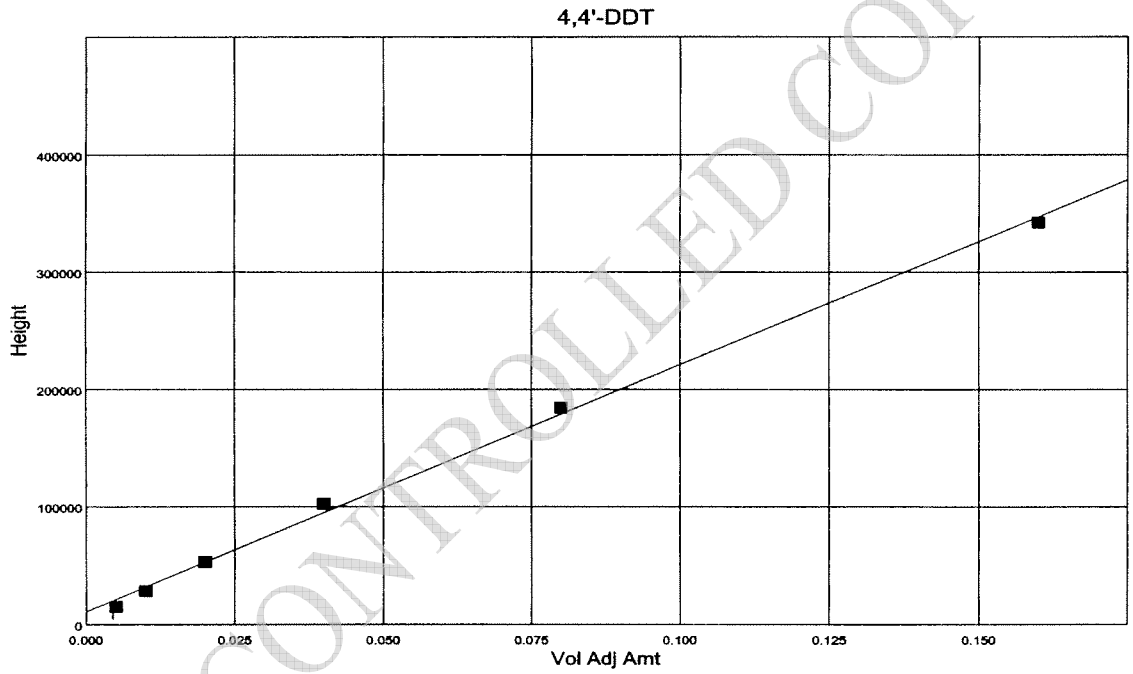
UNCONTROLLED COPY

Figure 2
DDT Calibration Curve Example

```
Fit Analysis Output For Method File: "C:\TC4\GC158\PE04304A.MTH"
Component Name: "4,4'-DDT"
Date: 5/3/04      Time: 15:52

Curve Parameters:

  Curve #1 :  1st Order
              Weighting Factor  =  1.0 (No Weighting)        r2  =    0.997960
              Calibration Curve =  (11057.842158) + (2099938.639920)X
```

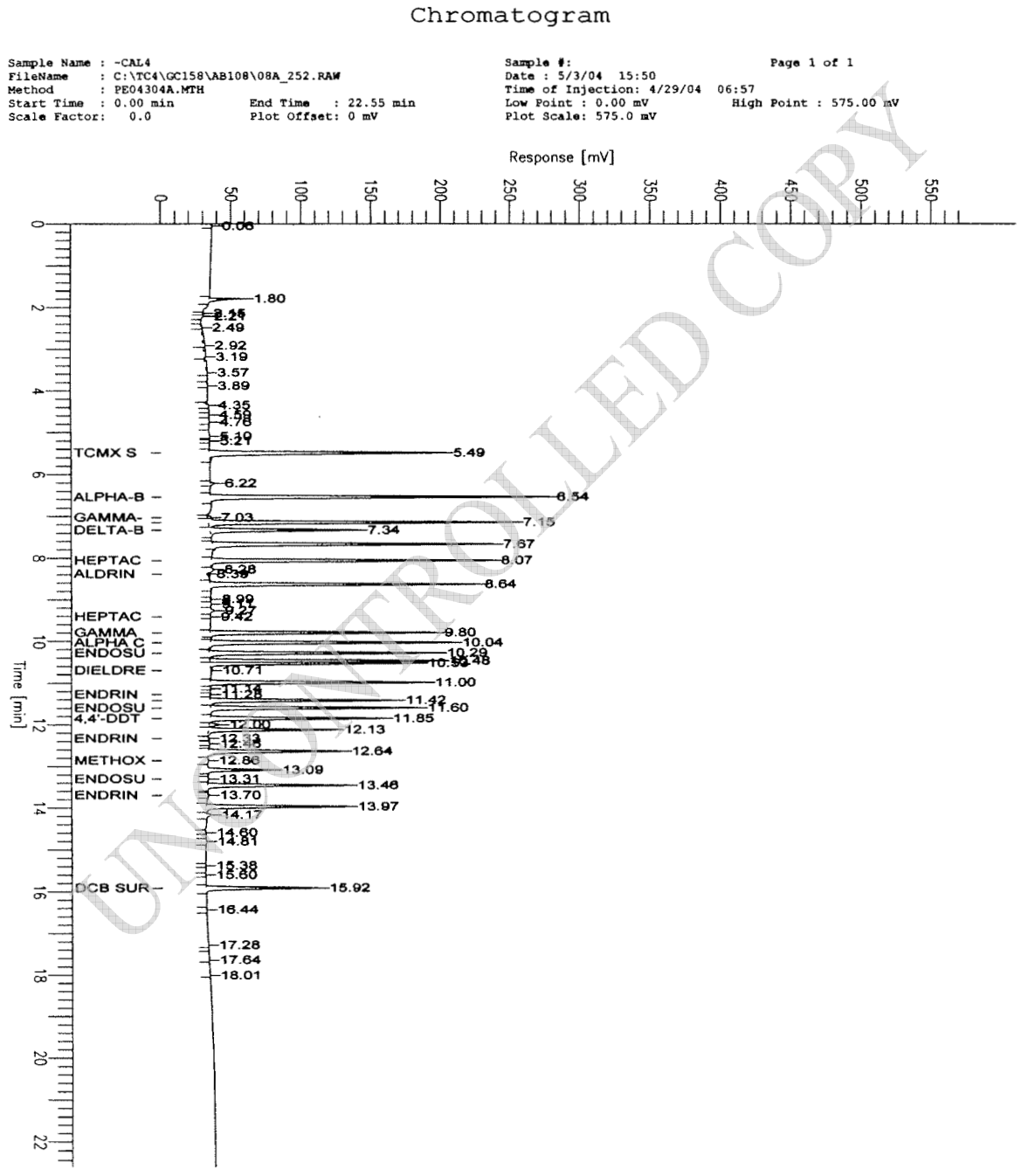


Level Name	Observed X-Value	Calculated X-Value	Delta	%Diff.	Observed Y-Value	Calculated Y-Value	Delta	%Diff.
1	0.005000	0.002087	0.002913	139.534	15441.228	21557.535	-6116.308	-28.372
2	0.010000	0.008480	0.001520	17.931	28864.284	32057.229	-3192.945	-9.960
3	0.020000	0.020167	-0.000167	-0.828	53407.121	53056.615	350.506	0.661
4	0.040000	0.043679	-0.003679	-8.423	102781.740	95055.388	7726.352	8.128
5	0.080000	0.082683	-0.002683	-3.245	184687.138	179052.933	5634.204	3.147
6	0.160000	0.157904	0.002096	1.327	342646.215	347048.025	-4401.810	-1.268

UNCONTROLLED COPY

Approved By: 1-29-10 **QA Officer** **Approved By:** 1/29/10 **Area Supervisor**

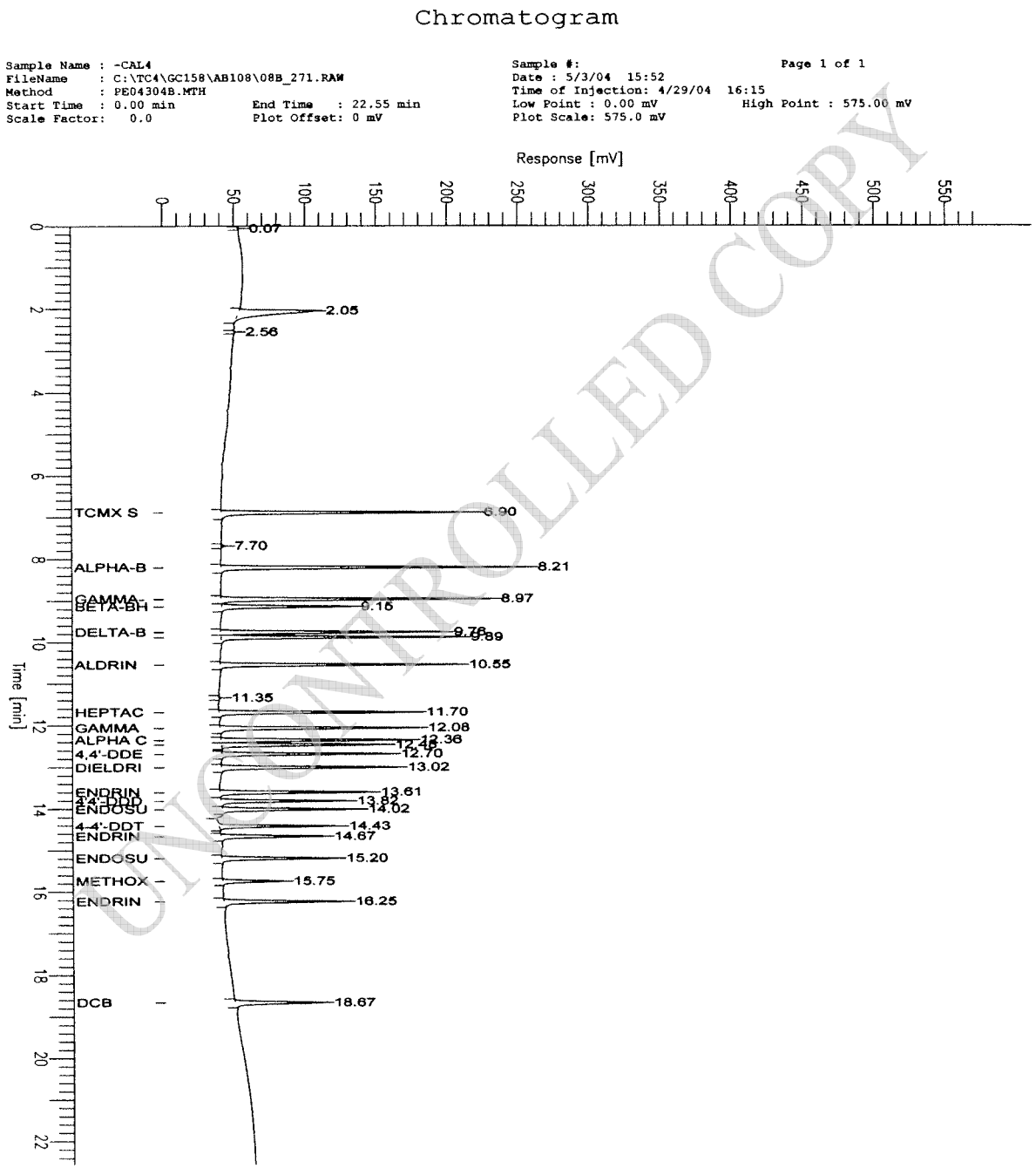
Figure 3
Pesticide Chromatogram On RTX CLP Column



Approved By: 1-29-10 **Approved By:** 1/29/10

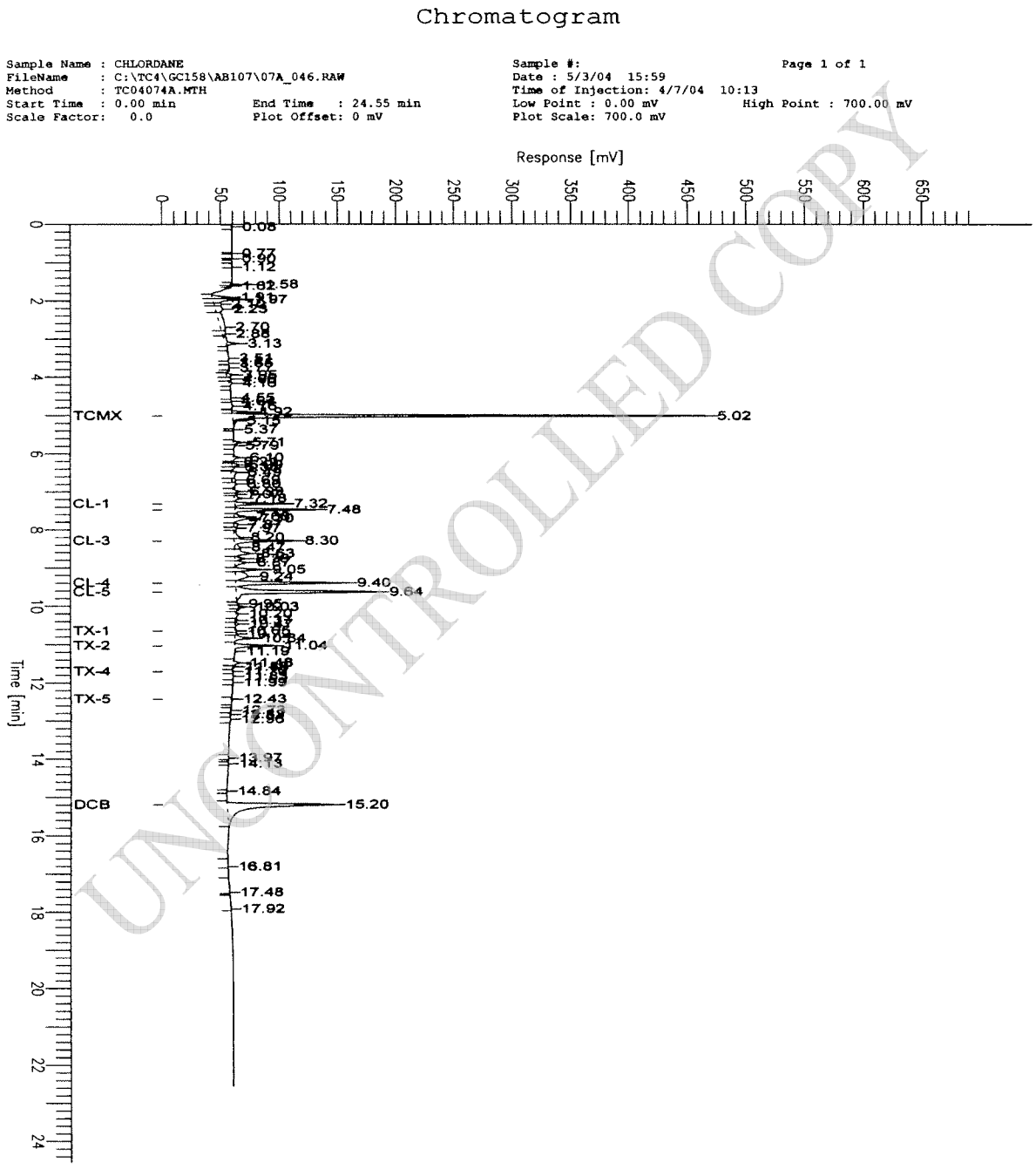
QA Officer **Area Supervisor**

Figure 4
Pesticide Chromatogram on RTX CLP2 Column



UNCONTROLLED COPY

Figure 5
Chlordane Multi-Component Pesticide Chromatogram



UNCONTROLLED COPY

Approved By: pm 1-29-10 **QA Officer**

Approved By: JMS 2-10-10 **Area Supervisor**

Figure 6
Data Review Report Example

TriMatrix Laboratories, Inc.

Data Review Report [redacted]
Sequence = 7102946 Page 1 of 21 on 12/21/2007 at 5:45

SampleID	Analysis	IResult	Dfin	FResult	FMRL	Qualifier	Recovery	RPD	Analyzed	Analyst	Batch
0710183-24	8081A PESTs (master list)	0.0695	80	11	0.80	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.00		S009	-cl		10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.00		S009	-cl		10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	2000	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	2000	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0869	40	7.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.085	40	6.8	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0722	80	12	0.80	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0678	20	2.7	0.20	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0681	40	5.4	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0642	80	10	0.80	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0688	40	5.5	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0688	40	5.5	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	400	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0903	40	7.2	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0693	20	2.8	0.20	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0731	80	12	0.80	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0959	80	15	0.80	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0	40	0.0	0.40	S009			10/29/2007 1:29	DJM	0712971
0710183-24	8081A PESTs (master list)	0.0686	80	11	0.80	S009			10/29/2007 1:29	DJM	0712971

UNCONTROLLED COPY

Figure 7
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0712971 Page 1 of 1

Printed: 12/21/2007 5:50:09PM

Semivolatiles GC, Water, 3510C Liquid-Liquid Extraction

Surrogate #1 = 7070019 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0710183	8081A PESTs (master list)	0710183	8081A MDEQ Pests	0710183	8081A APP IX Pests

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0712971-BLK1		Oct-25-07 18:18	ASC	1000	2	1000				BLANK	
0712971-DUP1		Oct-25-07 18:18	ASC	1000	2	1000	0710183-24			DUPLICATE	
0712971-DUP2		Oct-25-07 18:18	ASC	1000	2	1000	0710183-25			DUPLICATE	
0712971-BS1		Oct-25-07 18:18	ASC	1000	2	1000		7090392	100	LCS	
0710183-24	A	Oct-25-07 18:18		1000	2	1000				[redacted]	Added for BatchQC in: 0712971
0710183-24	A	Oct-25-07 18:18	ASC	1000	2	1000				[redacted]	[redacted]
0710183-24	A	Oct-25-07 18:18		1000	2	1000				[redacted]	Added for BatchQC in: 0712971
0710183-25	A	Oct-25-07 18:18	ASC	1000	2	1000				[redacted]	[redacted]
0710183-25	A	Oct-25-07 18:18		1000	2	1000				[redacted]	Added for BatchQC in: 0712971
0710183-25	A	Oct-25-07 18:18		1000	2	1000				[redacted]	Added for BatchQC in: 0712971
0712971-BLK2		Oct-31-07 15:18	KB9	1000	2	1000				BLANK	
0712971-DUP3		Oct-31-07 15:18	KB9	1000	2	1000	0710183-30			DUPLICATE	
0712971-DUP4		Oct-31-07 15:18	KB9	1000	2	1000	0710183-36			DUPLICATE	
0712971-BS2		Oct-31-07 15:18	KB9	1000	2	1000		7080865	200	LCS	chlordane
0712971-BS3		Oct-31-07 15:18	KB9	1000	2	1000		7070687	200	LCS	toxaphene
0710183-30	A	Oct-31-07 15:18	KB9	1000	2	1000				[redacted]	Added for BatchQC in: 0712971
0710183-30	A	Oct-31-07 15:18	KB9	1000	2	1000				[redacted]	Added for BatchQC in: 0712971
0710183-30	A	Oct-31-07 15:18	KB9	1000	2	1000				[redacted]	GOOD WP Total Chlordane
0710183-36	A	Oct-31-07 15:18	KB9	1000	2	1000				[redacted]	Added for BatchQC in: 0712971
0710183-36	A	Oct-31-07 15:18	KB9	1000	2	1000				[redacted]	GOOD WP Toxaphene
0710183-36	A	Oct-31-07 15:18	KB9	1000	2	1000				[redacted]	Added for BatchQC in: 0712971

Comments:	Analyst Initials:

bch_TriMatrix.rpt

UNCONTROLLED COPY

Approved By: JM 1-29-10 **QA Officer**

Approved By: JMS 2-10-10 **Area Supervisor**

Figure 8
Instrument Run Log Example

TriMatrix Laboratories, Inc.

Instrument 199 HP6890	Date: 1/10/08	Sequence #: A816	Date Archived:	Analyst:
Instrument Settings / Injection Volume	GC Program	Quantitation Information / Breakdown	Routine Maintenance Items	
Column Type: STXCLP / STXCLP2	Initial:	Curve Date:	New Column Date:	
Injection Volume: 1uL / 2uL	Hold:	Curve Type: Regression or Average CF	Column Clipped: Yes / No	
Injector / Detector: 210 / 320	Rate:	DDT % Breakdown <15%: YES / NO	New injection Port Liner: Yes / No	
Method: 8081 / 8082 / 608 / 608.2 / 8011	Final:	Endrin % Breakdown < 15%: YES / NO	New Septa: Yes / No	
Default Temperature Program: Yes / No	Hold:		New Syringe: Yes / No	

Run ID	File ID	Injection Time	Analysis Method	Client	Matrix	Dilution	ICV/CCV Check (85-115)	Sample Notes, Standard Numbers, Analytical Batch Information, etc.
PEM	147	12:00	8082A,B		QC		PASS	7110868
Inst BLK	148	12:35						
Tox ICV	149	13:11						8020476
P1	150	13:47						8010136
P2	151	14:23						135
P3	152	14:58						134
P4	153	15:34						133
P5	154	16:10						132
P6	155	16:45						8010108
PBCV	156	17:21						8010143
PEM	157	17:57						7110868
0802167-03	158	18:32			WTR			
↓ -01	159	19:08						
0801577-BSD	160	19:44						
-BS2	161	20:19						
↓ -BS1	162	20:55			↓			

file: 199_RUN.XLS

page: 15 of 50

revision: 1.1

UNCONTROLLED COPY

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Gasoline Range Organics (GRO) by Gas Chromatography

SW-846 Method 8015B

UNCONTROLLED COPY

APPROVALS:

Area Supervisor: Diane Van Male Date: 8-21-09
Diane L. VanMale

QA Officer: Date: 8-20-09
Tom C. Boocher

Operations Manager: Date: 8/21/09
Jeff P. Glaser

Procedure Number: GR-03-121
Revision Number: 3.4

Date Initiated: 2/1/94
Effective Date: 8/25/09

Date Revised: 8/20/09
Pages Revised: All

By: Diane L. VanMale
Total Number of Pages: 25

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is used to determine gasoline range organics in waters, soils and wastes, by purge-and-trap gas chromatography (GC) with flame ionization detection (FID).

1.2 Analysis is restricted to analysts qualified in the use of gas chromatography operation and hydrocarbon interpretation. Each analyst must have on record, training and qualification through demonstration of capability studies.

1.3 Reporting limits are as follows:

1.3.1 Waters, 0.1 mg/L

1.3.2 Low-level soils, 0.10 mg/kg (dry weight)

1.3.3 Methanol-preserved soils, 5.0 mg/kg (dry weight)

1.3.4 Waste, 50 mg/kg (wet weight)

1.4 Instrumentation includes a photoionization detector (PID) in series with the FID, allowing simultaneous volatile aromatics analysis, in accordance with TriMatrix SOP GR-03-105.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Method for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Update III, Revision 2, December 1996, Method 8015B

2.2 *Test Method for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Update II, Revision 1, July 1992, Method 8015A

3.0 SUMMARY OF PROCEDURE

3.1 Samples are concentrated by purge and trap, and desorbed to GC-FID for analysis. Gasoline range organics are separated within a specified retention time range by the GC column and temperature programming.

3.2 Detection is by Flame Ionization (FID). Quantification is based on the FID response to commercially prepared gasoline fuel standards.

4.0 PARAMETER OR COMPOUND LIST

4.1 Gasoline components of interest elute between C_6 and C_{10} hydrocarbons. The GRO retention time window is defined as all peaks between 2-methylpentane and 1,2,4-trimethylbenzene. Refer to Section 16.2.

UNCONTROLLED COPY

Note: The GRO definition, calibration and quantitation will vary by state. In cases of analytical conflict, the state method must supercede this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-04-105 *Closed System Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples,* latest revision

5.2 TriMatrix SOP GR-03-124, *Volatile Corrective Actions,* latest revision

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal,* latest revision

5.4 TriMatrix SOP GR-03-105, *Volatile Organic Compounds (VOC's) by Purge and Trap Capillary Column Gas ChromatographyPID/ELCD in Series 601/602/8021B*, latest revision

5.5 TriMatrix SOP GR-10-123, *Element Data Transfer and Review*, latest revision

5.6 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 At high concentration, mineral spirits, jet fuel, kerosene, diesel fuel, fuel oil, and stoddard solvent, elute peaks within the GRO retention time window. Other organics, including chlorinated solvents, ketones and ethers are also detectable. However, quantitation must include any peak that elutes from such interference, within the retention time window.

6.2 Samples can be contaminated by diffusion of volatiles through vial septa. A laboratory reagent water trip blank must be prepared and carried through collection, tranport, storage and sample handling.

6.3 Carryover can occur when high-level samples are analyzed. To reduce carryover, syringes and purge vessels must be rinsed between runs with reagent water or methanol. Highly concentrated samples must be followed by the analysis of at least one reagent blank. Sample analysis may not continue, until a clean blank is run.

6.4 If a purge vessel becomes fouled with soluble materials, suspended solids, high-boiling organics or organohalides, wash with detergent then rinse with laboratory reagent water and dry in a 105° C oven. Do not use a fouled purge vessel before cleaning.

6.5 The trap and other instrument components are also subject to contamination. Baking the trap and purging the system is frequently required.

7.0 SAFETY PRECAUTIONS

UNCONTROLLED COPY

7.1 The toxicity and carcinogenicity of reagents used in this procedure has not been precisely defined. All must be treated as a potential health hazard, and chemical exposure minimized. A laboratory coat, disposable gloves and approved safety glasses must be worn when working with reagents, solvents and standards. Approved safety glasses are required in the volatiles laboratory, except when entering data at computer terminals.

7.2 A reference file of Material Safety Data Sheets (MSDS) is available to all personnel on the intranet library drive.

7.3 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Collect aqueous samples in duplicate without headspace, in 40 mL contaminant-free borosilicate glass screw-cap vials with PTFE-lined septa. Vials must contain enough 1:1 HCl (v/v) to lower the pH to <2.0 when filled.

8.1.1 All vials provided by TriMatrix contain HCl preservative and must not be rinsed before filling. Fill with sample carefully, until a meniscus forms at the vial rim (almost to overflowing). Cap immediately.

8.1.2 To minimize analyte loss, fill vials so no bubbles pass through or are trapped in the sample after the vial is filled and sealed. After capping, invert to confirm no headspace or bubbles are present. If a vial contains bubbles larger than 5-6 mm, add additional sample or discard and fill another. Maintain samples at 0 – 6° C after collection, and during transportation to the laboratory. At the laboratory, store at 0 – 6° C and analyze within 14 days.

8.2 Bulk soils must be collected without headspace or preservative in contaminant-free 60 mL or 125 mL glass jars with PTFE-lined caps. Maintain samples at 0 – 6° C after collection and during transportation to the laboratory. At the laboratory, store at 0 – 6° C and analyze within 14 days. Refer to Section 13.2 for further soils preparation.

Note: Low-level bulk soil analysis is limited to concentrations less than 200 ug/kg. Methanol extraction of bulk soils must be used for samples exceeding 200 ug/kg.

Note: Bulk soils for analysis under Ohio VAP may only be collected if the expected total petroleum hydrocarbon (TPH) concentration exceeds 200 ug/kg unless reporting under method 8015A-modified.

8.3 Alternatively, soils may be collected with EnCore™ samplers. If used, preserve samples in the field with sodium bisulfate (low-level analysis) or methanol (high-level analysis) or receive by the laboratory within 48 hours of collection. For sampling and preservation details, refer to TriMatrix SOP GR-04-105. A summary is as follows:

8.3.1 Field Preservation for samples with concentrations exceeding 200 ug/kg is as follows:

UNCONTROLLED COPY

8.3.1.1 Pack soil without headspace into a 5 g or 25 g EnCore™ sampler. Cap with the stainless steel o-ring cap.

8.3.1.2 Transfer from the EnCore™ to a tared 40 mL vial containing 5 mL of sodium bisulfate or to a 60 mL tared sample jar for methanol preservation. Quickly add 25 mL of methanol to the 60 mL jar and close immediately.

8.3.1.3 Store and transport at 0 – 6° C.

8.3.1.4 On arrival at the laboratory, record the temperature and each sample container mass. Proceed to Section 8.3.2.6 unless samples are unpreserved.

8.3.2 Laboratory Preservation

8.3.2.1 Pack soil in EnCore™ samplers as outlined in Section 8.3.1.1.

8.3.2.2 Store at 0 – 6° C until analysis.

8.3.2.3 If not preserved in the field, samples must be preserved at the laboratory or freeze to at least -7° C within 48 hours of collection.

8.3.2.4 To preserve EnCore™ soils, transfer to a tared 40 mL vial or to a tared 60 mL glass jar by extruding with the supplied push rod. Record the soil/container mass to the nearest tenth of a gram using a top-loader balance.

8.3.2.5 Quickly add an equal volume of sodium bisulfate solution or methanol to obtain a 1:1 soil/preservative ratio.

8.3.2.6 For methanol-preserved samples, shake the jar for two minutes and let settle.

Note: Michigan samples require a 20-minute sonication step after shaking.

8.3.2.7 Once particulates settle out, decant by pouring into a 4.0 mL glass screw-cap vial, with a PTFE-lined septum cap. Do not use a pipet.

8.3.2.8 Label and store at 4° C in the volatiles laboratory soils refrigerator, until analysis.

8.3.3 To provide consistency in sample/preservative contact time, the preservative fluid must be decanted within 24 hours of receipt. However, if samples arrive on a weekend or holiday, decanting will be done the following business morning.

8.4 All soil samples must be analyzed within 14 days of collection.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

UNCONTROLLED COPY

9.1 Gas Chromatography System (refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications):

9.1.1 Autosampler: Dynatech PTA-30 w/s autosampler

9.1.2 Concentrator: Tekmar LSC-2000 concentrator

Conditions Used:
Trap ..... Supelco Trap K Vocarb 3000
Purge Gas ..... Helium at 40 mL/minute
Purge ..... 11 minutes at ambient temperature
Dry Purge ..... 2 minutes
Desorb Preheat ... 245° C
Desorb ..... 2 minutes at 250° C, gas flow at 15-20 mL/minute
Bake ..... 8 minutes at 260° C

9.1.3 Gas Chromatograph: A Hewlett Packard 5890 II, equipped with Flame Ionization Detector (FID) and Photoionization Detector (PID) in series

Conditions Used:
Injector Temp ..... 180° C
FID Detector Temp ..... 220° C
Hydrogen Flow ..... 35 mL/minute
Air Flow ..... 165 mL/minute
Make up flow ..... Helium at 20 mL/minute
PID Detector Temp ..... 220° C

NOTE: Gasoline range organics (GRO) must be quantitated by FID only. PID analysis for volatile aromatics is beyond the scope of this procedure (Refer to TriMatrix SOP GR-03-105).

9.1.4 GC Column: 30m x 0.53 mm ID, J&W Scientific DB-624, 3 micron film thickness

Conditions Used:
Temperature Program ..... 35° C, then 6.5° C/minute to 110° C, then 20° C/minute to 220° C, hold 2 minutes
Carrier Flow ..... Helium at 10 mL/minute

Note: Conditions may be altered to optimize resolution in the chromatography.

9.1.5 PE Nelson Turbochrome Data Acquisition Software (refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer specifications associated with the analytical instrument)

9.2 Glassware and Hardware

9.2.1 Balances:

UNCONTROLLED COPY

9.2.1.1 Analytical balance: Capable of weighing to the nearest 0.0001 g, used for standards

9.2.1.2 Top-loading balance: Capable of weighing to the nearest 0.1 g, used for samples

9.2.2 Ultrasonic bath

9.2.3 Sample containers:

9.2.3.1 Water samples: Borosilicate glass VOA screw-cap vials with PTFE-lined silicone septa caps, 40 mL

9.2.3.2 Soil samples: Glass jars with PTFE-lined caps, 4 oz or 8 oz

9.2.4 Borosilicate glass screw-cap vials with PTFE-lined septa caps, for bulk soils requiring methanol extraction in the laboratory, 20 mL

9.2.5 Syringes: Luerlock, glass, gas-tight, 5.0 mL

9.2.6 Glass screw-cap vials with PTFE-lined septa caps, for methanol extract storage, 4.0 mL

9.2.7 Volumetric flasks: 10 mL, 50 mL, 100 mL, 500 mL, 1000 mL

9.2.8 Microsyringes: 1.0 µL, 5 µL, 10 µL, 25 µL, 100 µL, 250 µL, 500 µL, 1000 µL

9.2.9 Disposable pipettes: Pasteur, 2 mL

9.2.10 Spatula: Stainless steel

9.2.11 Refrigerator: 0 – 6° C

9.2.12 pH test strips

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Due to its simplicity, FID maintenance is seldom required. The jet, collector electrode and detector body may need cleaned or replaced due to corrosion or chemical deposits. Refer to the FID manual for specific instructions.

10.2 High-boiling compounds can adversely affect chromatography. If a column becomes contaminated, clipping the first 6 inches from the injection end should restore performance.

10.3 Concentrator

10.3.1 Clean or replace purge vessels as necessary.

UNCONTROLLED COPY

10.3.2 Bake the trap out after high concentration samples are run. If baking does not eliminate carryover or trap performance deteriorates, replace the trap.

10.3.3 Check and record the purge flow weekly.

10.4 Autosampler

10.4.1 Fill the water reservoir and internal standards syringe daily.

10.4.2 Empty the waste reservoir daily.

10.4.3 Replace the sample syringe o-ring and valve block, if water leaks are observed.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory Reagent Water: Organic-free water purified using double-distilled reverse-osmosis feed water, followed by purging with nitrogen gas for 30 minutes before use.

11.2 Gasoline Fuel Standards: Certified solutions of unleaded gasoline are purchased at 10,000 ug/mL. A second source must be purchased as a Second Source Verification (SCV).

11.3 Gasoline Components Standard: A 10 component quantitation standard at 10,000 mg/L. Concentrations are listed in Table 1 (Attachment 23.1). This standard is used to define the GRO retention time window. Refer to Section 16.2.

11.4 Neat Compounds:

11.4.1 The surrogate used is ααα-trifluorotoluene. A stock solution at 10,000 ug/mL is prepared as in Section 12.3.2.

11.4.2 Fluorobenzene is used as the PID internal standard when analyzing GRO and volatile aromatics in the same run. A stock solution at 10,000 ug/mL is prepared as in Section 12.3.2.

11.5 Methanol: Purge-and-trap grade

11.6 Ottawa Sand: Baked at 400° C to remove contamination

11.7 Sodium bisulfate, 20%(w/v) in reagent water

12.0 STANDARDS PREPARATION

12.1 Input all laboratory standards to the laboratory information system (Element™). Standard description, vendor, part number, lot number, concentration, solvent, date opened, date expired and any other applicable

UNCONTROLLED COPY

information must be recorded. Each standard is given a unique ID number. Refer to Attachment 23.5 for an example.

12.2 Replace stock and intermediate standards within six months or sooner if quality control indicates a problem. Check intermediate solutions for evaporation and precipitation before using to recalibrate the GC. Store standards in the volatiles laboratory freezer at -5 to -20° C.

Note: No intermediate standard expiration date must exceed that of the stock standard from which it was prepared.

12.3 Stock Standards

12.3.1 Stock solutions for the Gasoline Components Standard and Gasoline Fuel Standard are purchased as certified solutions at a concentration of 10,000 ug/mL. Commercially prepared stock standards may be used at any concentration if certified by the manufacturer or an independent source. A separate-source stock standard (different vendor) must be purchased for preparation of the laboratory control sample (SCV).

12.3.2 Stock solutions may be prepared from neat chemicals, gravimetrically. The surrogate spike solution is prepared from neat compounds.

12.3.2.1 No concentration adjustment is needed if a neat chemical has greater than or equal to 96% purity. If purity is less than 96%, the percentage must be used in calculating subsequent solution concentrations.

12.3.2.2 Stock standard (10,000 mg/L per analyte) is prepared as follows:

12.3.2.2.1 Inject 0.5 g of each analyte (neat) into a tared 50 mL volumetric flask partially filled with methanol, on an analytical balance.

12.3.2.2.2 Record the mass of each analyte to the nearest 0.0001 g.

12.3.2.2.3 Dilute to volume with methanol, cap and invert three times for complete mixing.

12.3.2.2.4 To estimate approximate analyte volumes to inject into the volumetric, use the following equation:

$$(1/D_{analyte})*500\ \mu L = \text{analyte volume to inject } (\mu L)$$

where:
$1/D_{analyte}$ = Density of water in g/mL divided by analyte density, in g/mL

12.3.2.3 Transfer the stock solution into a 40 mL screw-cap vial. Store in the freezer, separate from samples and protect from light.

UNCONTROLLED COPY

12.4 Intermediate Standards

12.4.1 Intermediate standards are used to prepare calibration standards.

12.4.2 Gasoline and gasoline component solutions are prepared at 1,000 ug/mL by adding 100 µL of stock standard to 900 µL of methanol, in a mini-inert vial. Invert, and swirl to mix three times. Store in the volatiles laboratory freezer between -5 and -20° C. Refer to Attachments 23.2 and 23.3 for example chromatograms.

12.4.3 Fluorobenzene and ααα-trifluorotoluene Internal Standard/Surrogate are prepared at 250 ug/mL.

12.4.3.1 This solution is added to the PTA-30 W/S standards syringe.

12.4.3.2 Each sample must be spiked with 1.0 µL giving a concentration of 50 ug/L.

12.4.3.3 Prepare by transferring 1.25 mL of each stock standard in Section 11.4 using a gas-tight syringe to a 50 mL volumetric flask about ¾ full of methanol.

12.4.3.4 Bring to volume with methanol then cap and invert three times to mix.

12.4.3.5 Transfer to 20 mL vials and store in the volatiles laboratory GC freezer, at -5 to -20° C.

12.5 Working (Calibration) Standards

12.5.1 Calibration standards are prepared in water using the formula below:

$$V_s = (C_f V_f)/C_s$$

Where:
V_s = volume of intermediate standard injected, in µL
C_f = final concentration of working standard, µg/L
V_f = final volume of working standard, µL
C_s = concentration of intermediate standard, µg/L

Calibration standards must be prepared as needed and are not stored. Refer to section 14.1.

12.5.2 Calibration standards are prepared at a minimum of five concentrations. The lowest concentration must be at the reporting limit. The remaining calibration range must correspond to the expected sample concentration range, or must define the detector's linear range.

Note: All calibration standards must contain a constant methanol concentration, corresponding to the maximum methanol extract volume used for soil sample analysis (100 µL/5 mL water).

UNCONTROLLED COPY

12.5.3 Calibration standards are routinely prepared at 100, 200, 400, 600, 800 and 1,000 ug/L in water or sodium bisulfate solution appropriate to sample concentrations.

12.5.4 Calibration standard solution must be used to spike Blank (BS) and Matrix (MS/MSD) Spikes.

12.6 From the 5,500 ug/mL second-source gasoline stock standard in methanol, prepare the SCV as follows:

12.6.1 Water SCV: Use 5.0 µL of 5500 ug/mL to 50 mL water. The concentration is 550 ug/L

12.6.2 Soil SCV: Use 5.0 µL of 5500 ug/mL to 50 mL of a methanol extraction blank (10 g Ottawa Sand/10 mL methanol then 1.0 mL of the extract to 50 mL reagent water). The concentration is 2.75 mg/kg.

Note: If samples have been preserved with sodium bisulfate, include a soil SCV prepared with the reagent at a concentration matching sample concentrations.

13.0 SAMPLE PREPARATION

13.1 For aqueous samples, no preparation is necessary since the Dynatech PTA-30 w/s uses unopened 40 mL vials. Samples must come to ambient temperature before purging. The Dynatech takes a 5.0 mL aliquot from a vial, adds 1.0 µL of internal standard/surrogate then transfers to the sparger for purging. If a sample dilution is necessary, dilute into a volumetric flask and transfer to a new 40 mL vial. Dilutions must put concentration in the upper half of the calibration.

13.2 For bulk soils and sludges, methanol extraction is used as the default preparation. Waste samples are extracted if insoluble in methanol or diluted if soluble. After extracting, an extract aliquot is added to organic-free reagent water and purged at ambient temperature. Perform methanol extractions rapidly to avoid loss of volatiles as follows:

13.2.1 Before taking a subsample for extraction, mix any supernatant into the sample with a narrow metal spatula.

13.2.2 For sediments, soils or wastes insoluble in methanol, weigh 10 g (wet) into a tared 20 mL vial. Use a top-loading balance and record mass to the nearest 0.1 g.

13.2.3 Quickly pipette an equal volume of methanol into the vial (10.0 mL). Cap tightly and shake for two minutes.

13.2.4 After shaking, let the solids settle and decant by pouring into a 4 mL vial, with a PTFE-lined septum cap. Store methanol extracts in the soils refrigerator at 0 – 6° C until analysis.

Note: Ohio Voluntary Action Program (VAP) samples must have concentrations greater than 200 ug/kg to be analyzed as a bulk sample unless being reported under method 8015A-modified.

13.3 Refer to Section 8.3 for EnCore™ sample preparation.

UNCONTROLLED COPY

13.4 For all extracted samples, the following must be performed just before analysis.

13.4.1 Add 1.0 mL of methanol extract to a 50 mL volumetric flask ¾ full with reagent water using a gas-tight syringe.

13.4.2 Bring to volume and cap then invert to mix three times. Pour out the volume in the flask neck, and transfer the remainder to a 40 mL vial. Cap tightly.

13.4.3 Transfer to the PTA 30 W/S Autosampler for analysis. The Autosampler takes a 5.0 mL aliquot, adds 1.0 µL of internal standard/surrogate and transfers the aliquot to a sparge vessel for purging.

13.4.4 For larger extract dilutions, pipette a smaller extract volume to the 50 mL volumetric, adding additional methanol to total 1.0 mL of solvent added.

13.5 Low-level soils preparation

13.5.1 Some state or federal programs require lower reporting limits than achievable using methanol extraction. In such cases, soil samples must be analyzed directly using heated purge-and-trap. EnCore™-collected soil samples need no additional preparation once preserved and can be placed directly on the autosampler for analysis. Refer to Section 8.3 for EnCore™ sample preparation.

13.5.2 For bulk soils, weigh out 5.0 g of sample into a tared 40 mL vial, using a top-loading balance. Record the mass to the nearest tenth of a gram. Add 5.0 mL of reagent water, a PTFE-coated stir bar then quickly cap the vial. Place in the autosampler.

13.5.3 If any low-level sample concentration exceeds the calibration, re-analyze using methanol extraction according to Section 13.4.

Note: This procedure may not be used for Ohio Voluntary Action Program (VAP) samples unless being reported under method 8015A-modified.

14.0 CALIBRATION PROCEDURES

14.1 External calibration must be used for gasoline range organics analysis. Do not force the calibration through the origin.

14.2 Prepare an external calibration of six standards at concentrations indicated in Table 2 (Attachment 23.4) by combining appropriate intermediate standard volumes and methanol. The lowest concentration must be at the reporting limit. The others must correspond to the expected sample concentration range or must define the linear range of the detector.

14.3 Inject each calibration standard for analysis using purge-and-trap.

UNCONTROLLED COPY

14.3.1 Calibration is performed by comparison of total GRO area response (minus internal standard/surrogate areas) within a retention time window against the mass injected.

14.3.1.1 Turbochrome uses the coefficient of determination (r^2), rather than correlation coefficient (r). If linear regression is used, r^2 must be greater than 0.99.

14.3.1.2 If initial calibration does not meet r^2 criteria, then drop a point on either end of the curve and recalculate. The lowest point may only be omitted if the next lowest standard remains at the reporting limit. If the highest point is rejected, the calibration range will be reduced. More dilutions may be required.

Note: Nonlinear quadratic regression may not be used for the GRO calibration if it has been determined to be a linear regression.

14.3.2 Alternatively, a calibration factor (CF) may be used for the initial calibration. The CF is defined as the ratio of mass injected to response obtained. Calculate a CF for each GRO standard. If percent relative standard deviation (%RSD) of the average CF is less than 20%, linearity through the origin is obvious. Then, the average CF may be used in place of the linear regression curve.

$$CF_i = M_i/A_i \qquad CF_{avg} = (CF_1 + CF_2 + CF_3 + \ldots + C_i) / i$$

Where:

CF_i = Calibration factor
M_i = GRO mass purged, in mg
A_i = GRO area sum
i = calibration point, 1 through 6
CF_{avg} = Average calibration factor

14.4 The initial calibration must be verified each day, before samples are analyzed. Verification is a midpoint injection continuing calibration verification (CCV).

14.4.1 The CCV must be run immediately after the daily method performance blank, after every ten analyses and as the last analysis in an instrumental run.

14.4.2 Calculate percent difference if using a calibration factor or percent drift if using a regression curve as follows.

$$\text{Percent Difference} = \frac{CF_2 - CF_1}{CF_1} 100\%$$

Where:
CF1 = Average calibration, from the calibration
CF2 = Calibration factor from the CCV

UNCONTROLLED COPY

$$\text{Percent Drift} = \frac{OC - TC}{TC} \times 100$$

Where:
TC = Expected concentration of the CCV
OC = Observed concentration of the CCV

15.0 ANALYTICAL PROCEDURE

15.1 Gasoline Range Organics are injected into the gas chromatograph by purge and trap. For methanol extracts, a separate calibration must be used which includes 1.0 mL of methanol in each standard. The methanol concentration purged must be the same for all standards and samples.

15.2 Analyze an instrument/method performance blank consisting of laboratory reagent water, methanol and internal standard/surrogate to check for contamination. The concentration of any contaminant must be less than the reporting limit. If not, take corrective action in accordance with TriMatrix SOP GR-03-124.

15.3 Analyze the GRO retention time standard to determine the daily retention time window from 2-methylpentane to 1,2,4-trimethylbenzene. Refer to Section 16.2.

15.4 Analyze the six initial calibration standards, from the lowest to highest concentration. Review the calibration. The coefficient of determination (r^2) must be $\geq$0.99, if using a linear regression curve. If using the average CF, %RSD must be $\leq$20%. Take corrective action in accordance with TriMatrix SOP GR-03-124 if the calibration is unacceptable.

15.5 Analyze an instrument/method performance blank consisting of laboratory reagent water, methanol and internal standard/surrogate. The concentration of any contaminant must be less than the reporting limit. If not, take corrective action in accordance with TriMatrix SOP GR-03-124.

15.6 Analyze a second source verification (refer to Section 12.6). Recovery must be within 75-125% of the expected concentration. If not, take corrective action in accordance with TriMatrix SOP GR-03-124.

15.7 Analyze 10 samples or less. If a sample exceeds the calibration range, run a dilution. Follow any high-concentration sample with a blank analysis, to check for carryover.

15.8 Analyze a CCV. If percent drift/difference is greater than ±15%, perform corrective action as follows:

15.8.1 First, try re-making the CCV solution and re-running.

15.8.2 If this corrects the problem, continue with sample analysis.

15.8.3 If the re-analysis is out-of-control, run a new calibration and re-run all samples analyzed since the last acceptable CCV.

15.9 Repeat Sections 15.7 and 15.8 until all samples are analyzed.

UNCONTROLLED COPY

16.0 CALCULATIONS AND DATA HANDLING

16.1 Sample concentration is determined by calibrating GRO mass purged to the peak response summation between 2-methylpentane and 1,2,4-trimethylbenzene. Use the linear calibration curve or average calibration factor as determined in Section 14.3. Gasoline Range Organics is calculated as follows:

16.1.1 Aqueous Samples:

$$C_a = ((A_x * CF_{avg})/ V_s) * D \qquad \text{for average CF}$$

or

$$C_a = [(A_x\text{-}b)/(m * V_s)] * D \qquad \text{for linear regression}$$

Where:

C_a = Gasoline Range Organics, in mg/L
A_x = Gasoline Range Organics area summation
CF_{avg} = Average CF from the initial calibration, in mg/area
D = Dilution factor, in mL/mL (If no dilution is made, D = 1)
V_s = Sample volume purged, in L
b = Linear regression curve y-intercept, from the initial calibration
m = Linear regression curve slope, from the initial calibration

16.1.2 Methanol-Extracted Samples:

$$C_s = (A_x/W) * (V_t/V_i) * CF_{avg} * D \qquad \text{for average CF}$$

or

$$C_s = [(A_x\text{-}b)/(m * W)] * (V_t/V_i) * D \qquad \text{for linear regression}$$

Where:

C_s = Gasoline Range Organics, in mg/kg
V_t = Total extract volume, in µL.
V_i = Extract volume added for purging, in µL
W = Sample mass extracted, in kg (wet weight)
A_x = Gasoline Range Organics area summation
CF_{avg} = Average CF from the initial calibration, in mg/area
D = Dilution factor, in mL/mL (If no dilution is made, D = 1)
b = Linear regression curve y-intercept, from the initial calibration
m = Linear regression curve slope, from the initial calibration

UNCONTROLLED COPY

16.2 Retention Time Window and Pattern Recognition

16.2.1 Before establishing a GRO retention time window, be certain the instrumental system is at optimum operating conditions. Make three injections of gasoline components standard throughout the course of a 72-hour period. Serial injections over less than 72-hour period result in overly narrow retention time windows.

16.2.2 Calculate the standard deviation of the average retention time for 2-methylpentane and 1,2,4-trimethylbenzene.

16.2.2.1 A retention time window for component peaks is defined as plus or minus three times the standard deviation of the average retention time. The GRO retention time window is from the lower limit of the 2-methylpentane RT window to the upper limit of the 1,2,4-trimethylbenzene RT window.

16.2.2.2 If standard deviation for either compound is zero, use ±0.10 minutes as a retention time window.

16.2.3 Calculate the GRO retention time window daily by analysis of the GRO component standard.

16.2.4 Retention time windows must be calculated on each GC column and whenever a new GC column is installed. Retrain all retention time window data.

16.2.5 Identification of gasoline is complicated by volatility and weathering. Early-eluting volatile components might be missing. To minimize the effects of volatility and weathering, collect samples as soon as possible after a site is contaminated.

16.2.6 In some cases, a client may request site contaminant identification beyond the scope of this procedure. If volatile component mixtures are not present in the GRO run, analysis by methylene chloride extraction and direct injection is recommended. Comparison can then be made against any supplied fuels mixture.

16.3 Single-component organics including chlorinated solvents, ketones, and ethers are detectable within the GRO retention time window. The GRO result must include such compounds. However, narrate the sample if the GRO result includes a high concentration single-component volatile.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for data quality and for filling in the correct documentation. It is important to document analyses by correctly filling in, handing in and filing the correct paperwork. This is required for quality assurance and to provide clients with defensible data.

17.2 Refer to TriMatrix SOP GR-10-123 for entering sample data into LIMS (Element™).

17.3 If internal chain-of-custody is required, it is important that COC forms be filled in correctly. The entire time an analyst has sample possession must be accounted for.

UNCONTROLLED COPY

17.4 The carbon range used for GRO sample analysis must be included on the client report.

17.5 Laboratory Required Paperwork

17.5.1 All run, maintenance, CD backup and other logbooks must be filled in completely and correctly. Corrections must be made with a single initialed and dated lineout. Writeovers are not permitted. Blank lines in run logbooks must be Z'd out then initialed and dated.

17.5.2 All supporting instrument quality control hardcopy, including continuing calibration standards, blanks and retention time windows must be filed chronologically by instrument number in banker boxes.

17.5.3 All instrument documentation and raw data must be appropriately archived for easy storage and retrieval.

18.0 QUALITY ASSURANCE

18.1 A purged blank (BLK) must be analyzed before sample analysis. The blank must go through all preparation and analytical steps. The result must be less than the reporting limit before proceeding with sample analysis.

18.2 A continuing calibration verification standard (CCV) must be run before sample analysis begins and must be run after every 10 analyses. In addition, sample analysis must end with an acceptable CCV. If percent drift/difference is greater than ±15%, corrective action must be performed as follows:

18.2.1 First, try re-making the CCV solution. If this corrects the problem, continue with sample analysis.

18.2.2 If the re-analysis is still out-of-control, run a new calibration then re-run all samples beginning from the last acceptable CCV with one exception. If the CCV is out-of-control high and there are no reportable sample results, data may still be used with justifying narration on the report.

18.3 Calculate surrogate recovery for each analysis including samples and quality control. If recovery is outside laboratory established acceptance limits verify calculations, dilutions and surrogate solutions. Also, verify instrument performance. Re-analyze samples unless matrix interference is confirmed by matrix spike recovery.

18.3.1 A high recovery may be due to matrix co-elution. Examine the chromatogram to verify the surrogate peak is correctly identified and integrated. If surrogate recovery is out-of-control on the high side but samples are non-detect, no re-analysis is necessary (give justification in report). Re-run samples if there are positive sample results. Refer to TriMatrix SOP GR-03-124 for corrective action details.

UNCONTROLLED COPY

18.3.2 Low recovery may be due to matrix inhibition. Analyses with low surrogate recovery must be re-analyzed to verify matrix inhibition unless confirmed by sample duplicate or matrix spike runs.

18.3.3 Low recovery may also be due to poor purge efficiency because of a clogged sparge needle. Clean the sparge needle by sonicating in an ultrasonic bath. Re-analyze the sample.

18.4 Blank and Matrix Spikes

18.4.1 Analyze blank and matrix spikes at least once for each batch of up to 20 samples analyzed, per matrix. A blank spike must be analyzed every shift samples are run regardless of how few samples are run.

18.4.2 Until 20 matrix spikes, matrix spike duplicates and blank spikes have been analyzed, recovery will be evaluated against default limits of 50 – 150% and a maximum relative percent difference of 20%.

18.4.3 Quality control calculations are performed in Element™.

18.4.4 If recovery or duplication is not within acceptable limits, take corrective action in accordance with TriMatrix SOP GR-03-124, including sample re-analysis if necessary. Results being reported as estimated must include narration to that effect.

18.4.5 If a blank spike is out-of-control, immediately identify and correction the problem before further samples are run.

18.4.5.1 Every effort must be made to determine the reason for failure (mis-spiked, mis-purged or other issues) before corrective action is taken.

18.4.5.2 All samples run with the system out-of-control must be re-analyzed.

18.4.5.3 There is one exception. If the blank spike is out-of-control high and there are no reportable sample results data may still be used (with justification for using in the report).

18.5 Prepare the SCV as in Section 12.6.

18.5.1 Recovery must be within 75-125% of the expected value to validate the calibration standards before sample analysis.

18.5.2 If recovery is not within 75-125%, re-run the SCV once. If the second analysis fails, prepare a new calibration, run and repeat the SCV successfully. Re-run all samples from the last acceptable SCV.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

UNCONTROLLED COPY

19.1 Before any sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

19.1.1 Initial demonstration of Capability Study

19.1.1.1 Spike 25 µL of 1000 ug/mL of second source gasoline standard into each of four, 50 mL aliquots of water or water with 1.0 mL methanol extract from Ottawas Sand for soil samples.

19.1.1.2 Process through every step in the procedure, including acceptable quality control criteria.

19.1.1.2 Input all four results to the IDC spreadsheet located on the laboratory intranet library. Successful recovery must be within the SCV control limits and %RSD must be <20%.

19.1.1.4 If a failure occurs, locate and correct the source of the problem and repeat the study successfully. However, repeated failures will indicate a general problem with the procedure and/or techniques being used. If this occurs, locate and correct the procedure and/or techniques then repeat the study successfully.

19.1.1.5 Samples may not be analyzed by any analyst on any instrument until a demonstration of capability study has been successfully completed.

19.1.2 A continuing demonstration of capability (CDC) study must be performed annually by all analysts using this procedure. The CDC may be accomplished as follows:

19.1.2.1 By repeating the IDC study

19.1.2.2 By entering four consecutive and exclusively analyzed SCV results into the IDC spreadsheet

19.1.2.3 By analyzing an acceptable PT study sample

19.1.2.4 By exclusively performing a method detection limit study and using the last 4 results in the IDC spreadsheet.

19.2 A Method Detection Limit (MDL) study must be run annually in accordance with TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

UNCONTROLLED COPY

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) located on the laboratory intranet library when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with the chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Method for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Update III, Revision 2, December 1996, Method 8015B

22.2 *Test Method for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Update II, Revision 1, July 1992, Method 8015A

23.0 ATTACHMENTS

23.1 Gasoline Component Standards and Concentrations – Table 1

23.2 Example Chromatogram of Gasoline Fuel Standard

23.3 Example Chromatogram of Gasoline Components Standard

23.4 Standard Volumes Required for Calibration – Table 2

23.5 Standards Log Example

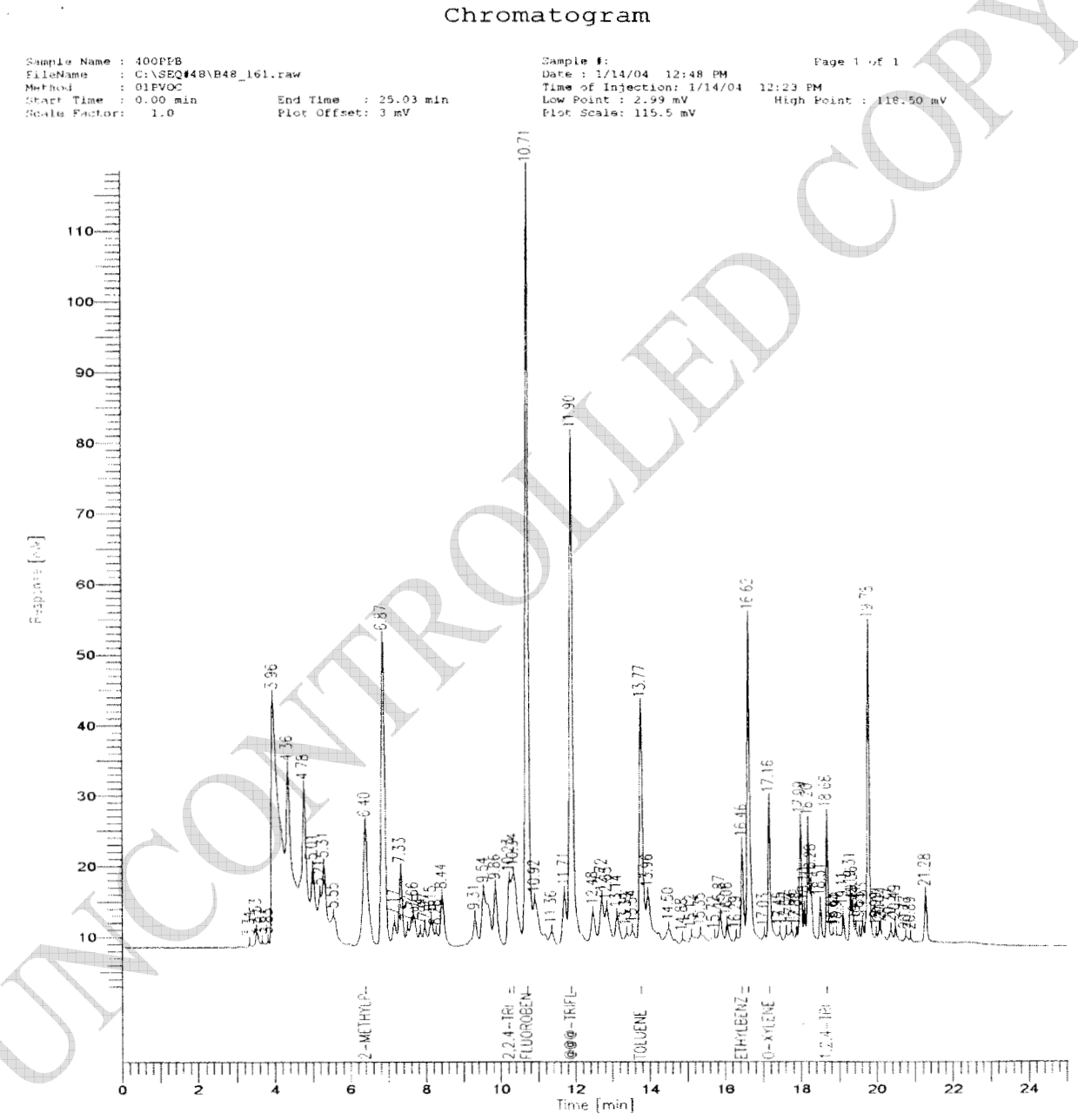
UNCONTROLLED COPY

Attachment 23.1
Table 1
Gasoline Component Standards and Concentrations

Component	Concentration, ug/mL
2-Methylpentane	1500
2,2,4-Trimethylpentane (iso octane)	1500
Heptane	500
Benzene	500
Toluene	1500
Ethylbenzene	500
m-Xylene	1000
p-Xylene	1000
o-Xylene	1000
1,2,4-Trimethylbenzene	1000
	10000 ug/mL Total

UNCONTROLLED COPY

Attachment 23.2
Example Chromatogram of Gasoline Fuel Standard



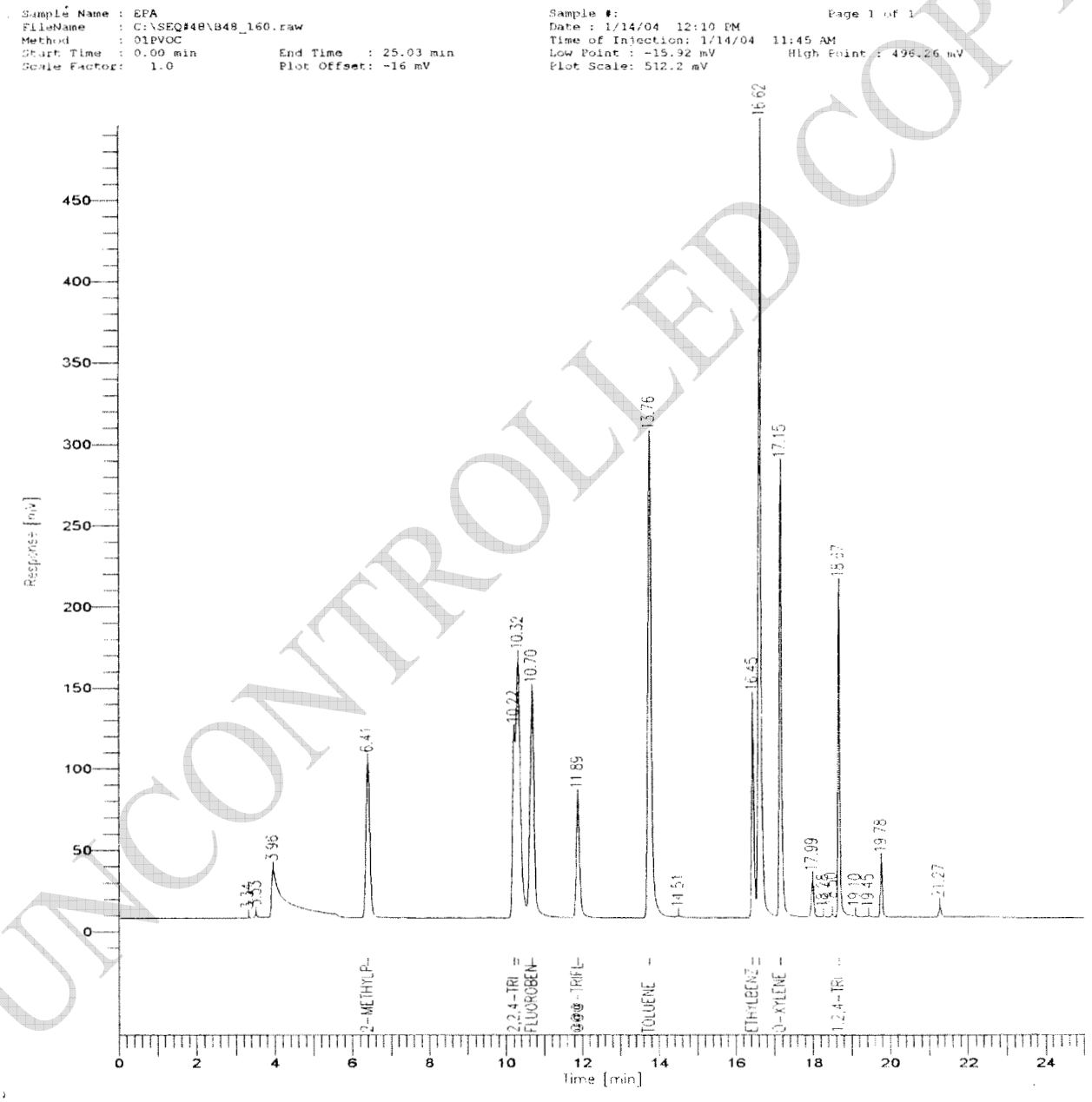
UNCONTROLLED COPY

Approved By: _____ 8-20-09 **Approved By:** _____ DLW 8-21-09

QA Officer **Area Supervisor**

Attachment 23.3
Example Chromatogram of Gasoline Components Standard

Chromatogram



UNCONTROLLED COPY

Approved By: _____ 8-20-09 **Approved By:** _____ DW 8-21-09
QA Officer **Area Supervisor**

Attachment 23.4
Table 2
Standard Volumes Required for Calibration

Concentration of Working Standard (ug/L)	Volume (µL) of Gasoline Fuel Standard 12.4.2	Additional Volume (µL) of Methanol Required
100	5	995
200	10	990
400	20	980
600	30	970
800	40	960
1,000	50	950
LCS-500	25	975

UNCONTROLLED COPY

Attachment 23.5
Standards Log Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

9040882

Description:	GRO CAL 3 040809	Expires:	Apr-27-09
Standard Type:	Calibration Standard	Prepared:	Apr-08-09
Solvent:	Solvent Lot #	Prepared By:	Laurel E. Wood
Final Volume (mls):	50	Department	Volatiles GC
Vials	1	Last Edit	Jun-22-09 10:35 by LEW

Calibration 02/12/09, EPAGRO51

Analyte	CAS Number	Concentration	Units
USEPA-8015B GRO Scan		0.4	ppm
GRO - 8015B (C6-C10)		0.4	ppm
Fluorobenzene	462-06-6	0.05	ppm
aaa-Trifluorotoluene	98-08-8	0.05	ppm

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
9010248	140 IS Mix 01/08/09	Jan-08-09	Laurel E. Wood	Apr-11-09	Apr-20-09 12:44 by LEW	0.01
9020454	87 Octane WKG A	Feb-12-09	Laurel E. Wood	Mar-12-09	Jun-22-09 10:35 by LEW	0.02

UNCONTROLLED COPY

Approved By: _____ 8-20-09 **QA Officer**

Approved By: _____ DLV 8-4-09 **Area Supervisor**

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Diesel Range Organics (DRO)

SW-846 Method 8015B

APPROVALS:

Area Supervisor: Janet M. Kudirka Date: 1/13/09
Janet M. Kudirka

QA Officer: _____ Date: 1-12-09
Tom C. Boocher

Operations Manager: _____ Date: 1/15/09
Jeff P. Glaser

Procedure Number: GR-03-122
Revision Number: 2.3

Date Initiated: 6/28/95
Effective Date: 1/20/09

Date Revised: 1/7/09
Pages Revised: All

By: Jeff P. Glaser
Total Number of Pages: 23

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 Analytes

1.1.1 This procedure is designed to measure Diesel Range Organics (DRO) in water and soil extracts. The analysis corresponds to an alkane range of C_{10} - C_{28} and a boiling point range of approximately 170° C and 430° C.

1.1.2 Diesel Range Organics measures mid-range petroleum products such as diesel or fuel oil. Components greater than C28 present in products such as motor oils or lubrication oils are detectable under conditions of the method. If, based on review of chromatogram, the presence of these product types is suspected, additional efforts may be performed including, but not limited to, analysis of additional reference materials. These additional efforts are not contained within this procedure.

1.2 Quantitation Limits

1.2.1 Quantitation limits are 0.2 mg/L for water and 6.7 mg/kg for soil.

1.3 Dynamic Range

1.3.1 Dilutions must be performed as necessary to put extract concentrations within the linear range of calibration. In general, the individual compound range is 12.5 ug/mL to 400 ug/mL in the final extract. This approximates 125 ug/mL to 4000 ug/mL of DRO.

1.4 Experience

1.4.1 This procedure is based on solvent extraction and gas chromatography (GC), and must be used by or under the supervision of experienced analysts.

1.4.2 Analysts must be skilled in chromatographic interpretation as a quantitative tool.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 2, December, 1996, Method 8015B, "Nonhalogenated Organics Using GC/FID"

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 2, December, 1996, Method 8000, "Determinative Chromatographic Separations"

3.0 SUMMARY OF PROCEDURE

3.1 One liter of water or thirty grams of soil are spiked with o-terphenyl surrogate and extracted with methylene chloride. Extracts are dried and concentrated to 1.0 mL, then injected into a capillary column gas chromatograph, equipped with a Flame Ionization Detector (FID). Quantitation is performed by comparing

total chromatographic area from alkanes C_{10} to C_{28}, including resolved and unresolved components, to the response of a diesel calibration.

4.0 PARAMETER OR COMPOUND LIST

Retention Time Standards	**Surrogate Standard**
Decane	Ortho-Terphenyl
Dodecane	
Tetradecane	
Hexadecane	
Octadecane	
Eicosane	
Decosane	
Tetracosane	
Hexacosane	
Octacosane	

Diesel Range Organics (DRO): All chromatographic peaks eluting between decane (n-C10) and octacosane (n-C28). Quantitation is based on direct comparison of the area within this range to the total area of a fuel standard calibration curve. Refer to Attachment 23.1 for an example diesel range organic chromatogram on a DB-5 column. Refer to Attachment 23.2 for an example retention time standard chromatogram on a DB-5 column.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-09-123, *EPA DRO Soil Extraction*, latest revision

5.2 TriMatrix SOP GR-09-124, *EPA DRO Water Extraction*, latest revision

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.4 TriMatrix SOP GR-04-101, *Semi-Volatile Organic Laboratory Corrective Action,* latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Organic compounds including, chlorinated hydrocarbons, phenols, and phthalate esters are detectable under conditions of this procedure. As defined by the method, the result includes these compounds if they fall within the DRO retention time range.

6.2 Interferences are reduced by washing all glassware with hot soapy water and rinsing with tap water, acetone, and then hexane. Immediately before use, the clean glassware is prerinsed with methylene chloride. Method performance blanks must be analyzed with each batch extracted or for every 20 samples, to demonstrate that reagents are free of contaminants.

6.3 High purity, pesticide-grade methylene chloride is used to minimize contamination from the solvent.

6.4 Contamination by carryover can occur whenever high and low level samples are sequentially analyzed. If an unusually concentrated sample is analyzed, a solvent blank analysis must be performed after the analysis to check for residual contamination.

6.5 Qualitative identification of diesel or other fuel types may be complicated by environmental processes such as weathering, biodegradation, or the presence of fuel mixtures.

7.0 SAFETY PRECAUTIONS

7.1 The toxicity or carcinogenicity of reagents used in this procedure have not been precisely defined. All chemicals must be treated as a potential health hazard. From this viewpoint, chemical exposure must be reduced to the lowest possible level by the proper use of hoods and personal protective equipment. A current file of Material Safety Data Sheets (MSDS) is available to all personnel on the laboratory intranet.

7.2 Solvents used in extraction and concentration of samples are highly toxic. Avoid all skin contact, vapor inhalation and ingestion. These solvents must only be used in a fume hood. Use protective clothing, disposable gloves and safety glasses when handling.

7.3 All analyzed sample extracts and expired standards must be properly disposed of, following the TriMatrix standard operating procedure GR-15-102 for laboratory waste disposal.

7.4 Analysts must comply with all procedures for health and safety, including those outlined in the TriMatrix Laboratory Safety Manual.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Water samples are collected in a 1 L glass container and acidified to pH 2 with HCl. Solids are collected in a core tube or glass jar. Samples are stored at 4° C ± 2° C from the time of collection until extraction. Extraction must be performed on water samples within seven days and soil samples within 14 days. All analyses must take place within 40 days, after extraction. Extracts are stored in the GC refrigerator at 4° C ± 2° C until analysis.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Gas Chromatograph: Hewlett Packard 5890 and 5890 series II gas chromatograph equipped with FID. Recommended conditions:

- Injector: 280° C
- Detector: 320° C
- Hydrogen Flow: 30 mL/minute
- Air Flow: 400 mL/minute
- Make-up: 20 mL/minute

9.2 Column

9.2.1 J&W DB-5 – 30 m X 0.53 mm ID, 0.5-micron film thickness or equivalent.

9.2.2 Temperature Program:

9.2.2.1 70° C hold for 1.0 minute, ramp 10° C/minute to 320° C, hold for 2.0 minutes.

9.3 Data System: PE Nelson Turbochrome Data Acquisition System

9.4 Autosamplers:

9.4.1 Leap A200SE

9.5 Note: FID must be used for DRO and other hydrocarbon measurements as described in this procedure. FID response is essentially the same for all hydrocarbons. Other detectors will not produce equivalent results.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Septa Replacement

10.1.1 Direct injection gas chromatographs are equipped with silicone septa (Supelco Thermogreen 10 mm, or equivalent), which eventually core and leak. These are replaced as necessary to avoid contaminating the inlet sleeve with rubber particles, and to eliminate bleed.

10.2 Inlet Sleeve Cleaning and/or Replacement

10.2.1 Inlet sleeves can become contaminated and restrict flow after accumulating sample debris and septum particles. Sleeves must be replaced periodically to minimize loss in resolution.

10.3 Column Clipping/Replacement

10.3.1 In addition to sleeve replacement and when resolution degrades, about 6" should be clipped off the front end of the column. When a column becomes too short for adequate resolution after clipping, it must be replaced.

11.0 CHEMICALS AND REAGENTS

11.1 Methylene Chloride, Hexane, Acetone: Pesticide grade or equivalent

11.2 Petroleum Fuel Product Standards – Refer to section 12.0 for preparation

12.0 STANDARDS PREPARATION

12.1 Stock Standard Solution: Prepare the following stock standards. Unless noted, all are prepared in the methylene chloride listed above. Standard preparation must follow guidelines in Method 8000.

12.1.1 Recommended Surrogate Standard: 2000 ug/mL ortho-terphenyl (OTP) stock. A working solution is made at 200 ug/mL by adding 1.0 mL of the stock standard to a 10 mL volumetric flask half full of methylene chloride. Dilute to volume, stopper, and invert three times to mix.

12.1.2 Diesel Component Standard: 20,000 ug/mL total concentration C_{10}-C_{28} even normal alkane compounds for establishing a DRO retention time window. A 1000 ug/mL standard is prepared by adding 500 uL of the stock standard to 10 mL in a volumetric, with methylene chloride.

12.1.3 Stock Laboratory Fortified Blank (LFB) – 50,000 ug/mL diesel #2. A working solution is made at 2,000 ug/mL in acetone (a water soluble solvent) by adding 1.0 mL of the stock standard to a 25 mL volumetric flask half full of acetone. Dilute to volume, stopper, and invert three times to mix.

12.1.4 Diesel Fuel #2, Fuel Oil #1, Kerosene-commercially manufactured stock standards at 50,000 ug/mL. Working standards are made at 4000 ug/mL by adding 2 mL of stock standard to a 25 mL volumetric flask half full of methylene chloride. Dilute to volume, stopper, and invert three times to mix.

12.2 All standards must be recorded in the semi-volatile Standards Logbook. Refer to Attachment 23.3 for an example of the standards log record. All standard vials must be labeled with the following information:

12.2.1 Standard name

12.2.2 Laboratory-assigned standard ID

12.2.3 Date made

12.2.4 Analyst initials

12.2.5 Solvent used

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 Standard preparation information is recorded in the semi-volatile Standards Logbook. Solutions are labeled with a two-letter prefix, GC, followed by a number depending upon logbook, page and line number. The nomenclature for standards naming will be as follows: lab area, book number, page number, line number. For example, GC2-4.8 would be from GC Standards Logbook 2, page 4, and line 8. Working standard solutions are stored in the Semi-volatile GC lab refrigerator at 4° C ± 2° C.

12.4 Shelf-life of stock standards is either one year from the date of preparation or the manufacturer's expiration date, whichever comes first. Working standard solutions expire six months from the date of preparation, or by the expiration of the stock standard, whichever comes first. Once a chemical or solution has expired, it

must be removed from the laboratory and properly disposed of. Monitor expiration dates of all chemicals and solutions. To avoid costly shipping, it is important to promptly order more chemical before the current supply is entirely consumed.

13.0 SAMPLE PREPARATION

13.1 Refer to TriMatrix SOP GR-09-123 (*EPA DRO Soil Extraction*) for extraction of soil samples.

13.2 Refer to TriMatrix SOP GR-09-124 (*EPA DRO Water Extraction)*, for extraction of water samples.

14.0 CALIBRATION PROCEDURES

14.1 Calibrate the GC with a minimum of five points using diesel fuel #2. Response of the standard is the sum of the peak area counts within the retention time window, for each concentration. The ratio of the area sum to concentration injected is the calibration factor (CF) and is calculated for each concentration, then averaged. If the percent relative standard deviation (% RSD) is less than 20% for the average CF, linearity through the origin can be assumed, and the average calibration factor can be used in place of a linear regression curve.

14.1.1 Calibration Factors (CF) for each calibration point are calculated using the following formula, then used to determine an average calibration factor:

14.1.1.1 $$CF_{stdx} = A_{stdx}/C_{stdx}$$

Where:
A_{stdx} = Peak area summation of a standard, adjusted to compensate for methylene chloride
C_{stdx} = Standard concentration number "x" in ug/mL, where x = 1 through 5

14.1.1.2 $$CF_{avg} = [CF_{std1} + CF_{std2} + CF_{std3} + CF_{std4} + CF_{std5}]/5$$

Where:
C_{stdx} = Standard concentration in ug/mL

14.1.2 Percent Relative Standard Deviation (%RSD) is calculated using the following formula:

$$\%RSD = [(SD_{avg})*100]/CF_{avg}$$

Where:
SD_{avg} = standard deviation of the average CF
CF_{avg} = average calibration factor for an analyte

14.1.3 Percent RSD must be ≤20% using at least 5 calibration points.

14.1.4 If %RSD for any analyte is greater than 20%, a regression curve must be generated by plotting peak area against concentration. A linear regression fit is used, without forcing through the

origin. To be acceptable, the correlation coefficient "r" must be 0.995 or higher, or the coefficient of determination (COD) "r^2" must be 0.990 or higher.

14.1.4.1 Linear regression requires five calibration standards and is calculated based on the following:

$$C_{ext} = (A_{DRO}-b)/a$$

Where:

A_{DRO} = Peak area summation in sample "s"
C_{ext} = Concentration of DRO in extract "ext"
a = Slope of the line (also called the coefficient of C) "s"
b = The intercept

14.1.5 When requested, samples will be qualitatively screened to identify a fuel type other than already listed, and a matching calibration curve run for quantitation. Whenever possible, calibration will be performed using the specific fuel contaminating a site. If it is not possible to identify a fuel, samples will be quantitated against fuel that best approximates the chromatographic fingerprint and retention time window.

14.1.6 **Note: A Diesel fuel #2 calibration will be used for all samples, unless otherwise requested.**

14.2 The average calibration factor or linear regression curve must be verified each working day by injection of an Initial Calibration Verification (ICV) standard and by analysis of subsequent Continuing Calibration Verification (CCV) standards, every ten sample injections. If calibration factors for these injections vary from the predicted response by more than ±15%, a new calibration must be prepared.

$$\text{Percent Difference} = \frac{CF2 - CF1}{CF1} \times 100\%$$

where:
CF1 = Average CF from the calibration curve
CF2 = Calibration Factor from ICV or CCV

14.3 The six points used to calibrate are 200, 250, 500, 1000, 2000, 4000 ug/mL. The lab reserves the right to change calibration concentrations, depending upon project requirements. The low calibration standard will determine the minimum reporting limit, and the high calibration standard will define the upper linear range.

14.4 Prepare a separate-source Laboratory Control Sample (LCS) to validate calibration standards accuracy. The LCS concentration should be equal to the midpoint of the calibration curve when prepared. Analysis recovery must be within 75-125% of the LCS expected value, to validate calibration and begin sample analysis. If recovery is not within 75-125%, the LCS must be reinjected. If the second injection fails, a new calibration curve must be prepared and validation repeated.

15.0 ANALYTICAL PROCEDURE

15.1 Instrument Set-up

15.1.1 Conditions (Recommended): Set helium column pressure to 4 mL/minute (±1), Air to 400 mL/minute (±15), Hydrogen to 30 mL/minute (±1), and make-up to 20 mL/minute (±1). Set column temperature to 70° C for 1.0 minute, increasing to 320° C at 10°/minute, then hold for 2.0 minutes. (Run time = 28.00 minutes). Set the FID to 320° C and injector to 280° C.

15.2 Retention Time Window Definition

15.2.1 Before establishing retention time windows, be certain the GC system is at optimum operating conditions. Make three injections of the diesel component standard throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

15.2.1.1 The retention time window for o-terphenyl is defined as plus or minus three times the standard deviation of the average retention time. The diesel fuel retention time window is based on the retention times of C10 and C28.

15.2.1.2 In those cases where the standard deviation for a particular peak is zero, the laboratory will use the average retention time ± 0.05 minutes as a retention time window.

15.2.2 The laboratory must calculate retention time windows for o-terphenyl and diesel fuel on each GC column, and whenever a new column is installed. Retention time studies must be retained in the laboratory.

15.3 Gas Chromatograph Analysis

15.3.1 Samples are analyzed by GC/FID only. The suggested injection volume is 1.0 uL, using the conditions established in 15.1.1.

15.3.2 A methylene chloride solvent blank must be run in each analysis sequence to determine the area generated by baseline bleed, prevailing over the 24-hour period of the sequence. This area is generated by projecting a horizontal baseline between the retention times of n-C_{10} and n-C_{28}. The area must be subtracted from the DRO area of all standards and samples run within the analysis period.

15.3.3 If initial calibration has been performed, verify by analysis of a mid-point ICV initially, followed by continuing calibration verifications (CCV) every ten sample injections, and at the end of the analytical sequence.

15.3.4 Calculate the ICV/CCV percent differences from the mean calibration factor, as in 14.2. If any calibration verification has a percent difference greater than ±15%, the instrument must be recalibrated.

15.3.5 Forward baseline projection must be used to integrate the DRO area count. (Valley-to-valley integration disregards the un-resolvable area of the chromatogram, which contributes

significantly to the DRO area count). However, valley-to-valley integration must be used for the surrogate.

15.3.6 Methylene chloride blanks must be run after samples suspected of being highly concentrated, to minimize carryover.

15.3.7 If product concentration exceeds the linear range of the calibration in the final extract, the extract must be diluted and reanalyzed. The individual compound range is 12.5 ug/mL to 400 ug/mL in the final extract. This is approximately equivalent to 125 ug/mL to 4000 ug/mL of diesel.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Quantitate using the average CF of the calibration, or a linear regression curve as specified in Section 14.1.

16.2 Use the equations below to calculate ug/mL of DRO in an extract:

16.2.1 Calculate an adjusted DRO peak area by subtracting out surrogate and methylene chloride areas:

$$A_{DRO} = A_{fb} - S_{vv} - B_{mc}$$

Where:
A_{fb} = DRO area using forward baseline integration
S_{vv} = Surrogate area using valley to valley integration
B_{mc} = Methylene chloride blank using forward baseline integration
A_{DRO} = The adjusted DRO area used for quantitation

16.2.2 For an average calibration factor, use the following equation:

$$C_{ext} = A_{DRO}/CF_{avg}$$

Where:
CF_{avg} = The average calibration factor from the initial calibration (refer to section 14.1)
C_{ext} = DRO extract concentration in ug/mL

16.2.3 For a linear regression curve, use the following equation:

$$C_{ext} = (A_{DRO} - b)/a$$

Where:
A_{DRO} = The adjusted DRO area used for quantitation
C_{ext} = DRO extract concentration in ug/mL
a = Slope of the line (also called the coefficient of C) "s"

b = The intercept

16.3 Next, calculate sample concentration by one of the following equations:

16.3.1 Water Samples:

$$C_{s\text{-}water} = (C_{ext} * V_e * DF)/V_w$$

Where:

V_e = Total extract volume in mL
DF = Extract dilution factor
V_w = Sample volume extracted in mL
C_{ext} = DRO extract concentration in ug/mL
$C_{s\text{-}water}$ = DRO sample concentration in mg/L

16.3.2 Soil samples:

$$C_{s\text{-}soil} = (C_{ext} * V_e * DF)/(V_s * PS)$$

Where:

V_e = Total extract volume in mL
DF = Extract dilution factor
V_s = Sample mass extracted in grams
C_{ext} = DRO extract concentration in ug/mL
$C_{s\text{-}soil}$ = DRO sample concentration in mg/kg
PS = Sample percent solids in decimal form (0.98 not 98%).

16.3.3 Use the method of external standards to calculate concentrations in all samples and spikes.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for data quality and also for filling in documentation correctly. It is important to document each analysis by correctly filling in, handing in, and filing paperwork. This is required for quality control purposes, and to provide clients with defensible data.

17.2 LIMS Reporting

17.2.1 When an analyst finishes running a sample batch, all data must be given to LIMS. Benchsheets must be filled in completely to ensure that results are reported correctly, and samples are associated with the right quality control. It is important that the quality control batch number from the extraction summary and analytical batch information from the 24-hour shift is filled in correctly. Dilution factors need added so reporting limits can be raised accordingly. All

positive hits must be recorded by crossing out the default reporting limit and writing down the final client result to the right of the reporting limit. This result must already have been through all necessary calculations, including the dilution factor. If a dilution is made to an extract and there is a positive hit, the elevated reporting limit must be written to the left of the default reporting limit. Surrogate results are reported by extract concentrations found, amount spiked, and percent recovery obtained.

17.2.2 If there are matrix spikes, the quality control benchsheet must also be handed in. In addition to section 17.2.1 requirements, quality control benchsheets report matrix spiking by the concentration spiked and percent recovery/percent difference for spike/spike duplicates. If matrix spike results are out-of-control due to extraction or matrix problems, the exclude (EXC) box must be checked to prevent biasing acceptance limit statistics.

17.2.3 An extracted Method Preparation Blank (MPB) must be handed in for every extraction batch. A daily blank (BLK) must be handed in with every 24-hour shift. Both sets of benchsheets need handed in. It is important to remember that no LFB can be handed in without first having handed in the associated MPB.

17.2.4 Internal chain-of-custody (COC) forms must be filled in correctly, when required. These are a very important part of the data package.

17.2.5 All LIMS benchsheets (including COC forms) must be placed in the correct colored folder and given to data entry. Blue folders containing BLK benchsheets must also include raw data quantitation reports and chromatograms.

17.3 Laboratory Required Paperwork

17.3.1 All run, maintenance, tape, and standard logbooks must be filled in completely and correctly. Corrections must only be made using line outs, not writeovers or scribbles. Blank lines in run logbooks must be Z'd out.

17.3.2 All ICV and CCV standard runs must be archived.

17.3.3 All LIMS documentation except for sample and quality control benchsheets, and raw data except for blanks and ICV/CCV standard runs, must be placed in the correct folder then given to the Semi-Volatile Laboratory technician. The technician must record the date, time and contents handed in.

17.4 Rounding and Significant Figures

17.4.1 Rounding is performed on final quantitated results only. All non-quality control results are rounded to 2 significant figures.

17.4.2 Quality control results are reported as whole numbers up to 3 significant figures.

18.0 QUALITY ASSURANCE

18.1 A method preparation blank (MPB) must be extracted with each extraction batch. The DRO concentration present in an MPB must be less than the reporting limit. If this limit is exceeded, samples with positive results must be re-extracted. If there is insufficient sample to re-extract or the holding time has expired, positive results must be qualified as estimated based on laboratory contamination.

18.2 Continuing calibration verifications (CCV) must be run before sample analysis begins, and after every 10 samples (refer to section 15.3.4). The analytical sequence must also end with a passing calibration verification. Acceptance limits for CCVs are ±15% difference from the calibration. If the CCV fails high, only samples with positive results require re-analysis. If the CCV fails low, all samples run before and after the CCV must be re-analyzed. If corrective action does not resolve the problem, a new initial calibration must be performed.

18.3 Calculate surrogate recovery for each extract analysis. If recovery or precision is not within acceptable limits, consult TriMatrix SOP GR-04-101 to determine when and how data is qualified.

18.3.1 High recovery may be due to a co-eluting matrix interference from the sample. Examine the chromatogram for evidence of co-elution. No corrective action is required in this instance.

18.3.2 Low recovery may be due to poor extraction efficiency. This should be verified by re-extracting the sample, if sample volume and hold times permit.

18.3.3 If surrogate recovery in a method performance blank (MPB) is below the lower control limit, only samples with failing surrogate recoveries will require re-extraction. If MPB surrogate recovery is above the upper control limit, no corrective action is required as long as sample surrogate recoveries are acceptable.

18.3.4 If surrogate recoveries are outside control limits in the MS/MSD, re-analysis is only required if LFB spike recoveries are also outside control limits. If the LFB is still out-of-control after re-analysis, all associated samples must be re-extracted.

18.4 Laboratory Fortified Blanks (LFB) and Matrix Spikes

18.4.1 Matrix spike and matrix spike duplicates are extracted at least every 20 samples, for each matrix. LFBs are extracted every 20 samples or each shift, for each matrix. Whichever is more frequent. This QC must be analyzed with the samples extracted

18.4.2 Until 15-20 Matrix Spikes (MS), Matrix Spike Duplicates (MSD), and LFBs have been analyzed, recoveries of each will be validated against default limits of 50-150%, and a duplication limit of 20% RSD.

18.4.3 To calculate recovery limits, calculate average percent recovery (R) and standard deviation (SD) of the average, by matrix. Express acceptable recovery as the interval from R – 3*SD to R + 3*SD. For example, if R = 90% and SD = 10%, recovery limits would be 60-120%. Precision limits for spike duplicates are calculated the same way except using average and standard deviation of relative percent difference. Limits must be updated at least annually.

18.4.4 Calculate percent recovery as follows:

$$\text{Percent Recovery} = \frac{|(\text{Aspk} - \text{Asmp})|}{\text{SPK}} \times 100$$

where:

Aspk = actual concentration found in the spiked sample, in mg/L or mg/kg
Asmp = amount found in non-spiked sample, in mg/L or mg/kg
SPK = amount spiked, in mg/L or mg/kg [(concentration of spike standard in ug/mL x mL spiked) / initial sample volume (mL) or mass (g)]

18.4.5 Calculate the relative percent difference as follows:

$$\text{Percent RPD (\%RPD)} = \frac{|(\text{SPK1} - \text{SPK2})|}{\left[\frac{(\text{SPK1} + \text{SPK2})}{2}\right]} \times 100$$

where:

SPK1 = Matrix Spike
SPK2 = Matrix Spike Duplicate

18.4.6 If recovery or precision is not within acceptable limits, the Corrective Action SOP GR-04-101 will determine when and how data is qualified.

18.4.7 If any LFB is out-of-control, the problem must be immediately identified and corrected before further samples are run. Failure of an LFB requires corrective action. Every effort must be made to determine the root cause of failure (for example: mis-spiking or mis-extracting). Once the cause is found, corrective measures must be taken

18.4.7.1 Failure of an LFB requires re-extraction of the sample batch. If re-extraction is not possible, qualify as follows:

18.4.7.1.1 If DRO in the LFB exceeds the upper recovery limit, positive results for associated samples in the batch are to be reported as "estimated".

18.4.7.1.2 All results less than the reporting limit are acceptable and need no qualification (LIMS qualifier 66).

18.4.7.1.3 If DRO recovery in the LFB is less than the lower control limit, positive results for every sample in the batch must be qualified as "estimated" and the reporting limit considered an approximation. A batch narrative must be written each time the LFB fails (LIMS qualifier 5).

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

19.1.1 Preparation and analysis of an initial demonstration of capability.

19.1.1.1 Spike 0.500 mL of 2,000 ug/mL diesel fuel #2 standard and 1.0 mL of o-Terphenyl surrogate spiking solution into each of four, 1000 mL aliquots of water or 30 g aliquots of Ottawa Sand. Process following every step in the extraction procedure. The IDC spiking solution must be prepared independently from standards used for quantitation.

19.1.1.2 Analyze the four aliquots following every step in the procedure.

19.1.1.3 Calculate average DRO recovery "x" in mg/L(or mg/kg), and standard deviation of the average "s" in mg/L for each analyte, using all four results.

19.1.1.3.1 Average recovery "x" must be within laboratory established limits, and relative standard deviation "s" must be $\leq$20%. If "x" and "s" meet these criteria, the demonstration of capability study is complete. The analyst and the instrument are authorized to run samples.

19.1.1.3.2 When an analyst fails to achieve acceptable results, Section 19.1.1.4 must be followed.

19.1.1.4 Locate and correct the source of the problem and repeat the study. Repeated failure however, may indicate a general problem with technique. If this occurs, locate the problem and correct the technique, then repeat the study. Samples may not be analyzed by any analyst or on any instrument until an IDC is successfully completed.

19.1.1.5 Demonstration of capability studies must be given to the Quality Assurance department for analyst training purposes.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A demonstration of capability study must be performed annually. A CDC may be accomplished by repeating the IDC study, by processing four consecutive LCSs run during the course of routine sample analysis on the IDC spreadsheet, or by submitting an acceptable PT result on the appropriate paperwork. LCSs and PT samples must have been run exclusively by the analyst to be used for a CDC study.

19.2 Method Detection Limit Studies

19.2.1 MDL studies must be performed annually on every instrument using this procedure. The MDL is defined as the minimum concentration of a substance that can be measured and reported with

99 percent confidence that the value is above zero. Actual reporting limits are derived from the MDL study. The minimum possible non-estimated reporting limit is equal to the concentration spiked in the MDL study, provided the MDL passes. Reporting limits achieved for any given analysis will vary depending on instrument sensitivity, matrix effects and dilutions. MDL studies must be completed for both water and soil matrices.

19.2.2 The procedure followed for a MDL study is based on the method given in 40 Code of Federal regulations, Part 136, Appendix B, latest revision.

19.2.3 Seven replicate analyses are performed using reagent water or clean sand, spiked with #2 Diesel at the estimated minimum reportable concentration (0.1 mg/L or 6.7 mg/kg). The last four of the seven water results can be used for an IDC or CDC study, if analyzed exclusively by one analyst.

19.2.4 Since a blank is required to calculate the measured level of DRO, seven separate blanks must be analyzed, one after each of the seven MDL samples. The average blank measurement is subtracted from each of the seven MDL results.

19.2.5 The standard deviation of the average found for the analyte(s) using all seven results is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

19.2.6 If the concentration spiked is between the calculated MDL and ≤ 5 times the calculated MDL, and there are no zero percent recoveries in the set of seven, the MDL is acceptable. If not, the MDL must be re-analyzed. If a study needs re-analyzed at a different concentration, the entire set of seven needs re-analyzed. If a study does not pass due to poor reproducibility on one of the results, only that result needs re-analyzed. Not more than one result may be rejected and re-analyzed however. All seven results do not need obtained in the same analytical batch.

19.2.7 If at any time the nominal reporting limit is above a client's or state's desired reporting limit, the calculated MDL may be used as the reporting limit, provided results are narrated. The narration must state that the reporting limit is based on the calculated MDL, and DRO when spiked at that level was not observed.

19.2.8 Refer to Attachment 23.6 for a Method Detection Limit Study Example.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix Laboratory SOP GR-15-102 (*Laboratory Waste Disposal)*, for laboratory waste disposal.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 2, December, 1996, Method 8015B, "Nonhalogenated Organics Using GC/FID"

22.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 2, December, 1996, Method 8000, "Determinative Chromatographic Separations"

23.0 ATTACHMENTS

23.1 Example Diesel Range Organic Chromatogram on DB-5 Column

23.2 Example Retention Time Standard Chromatogram on DB-5 Column

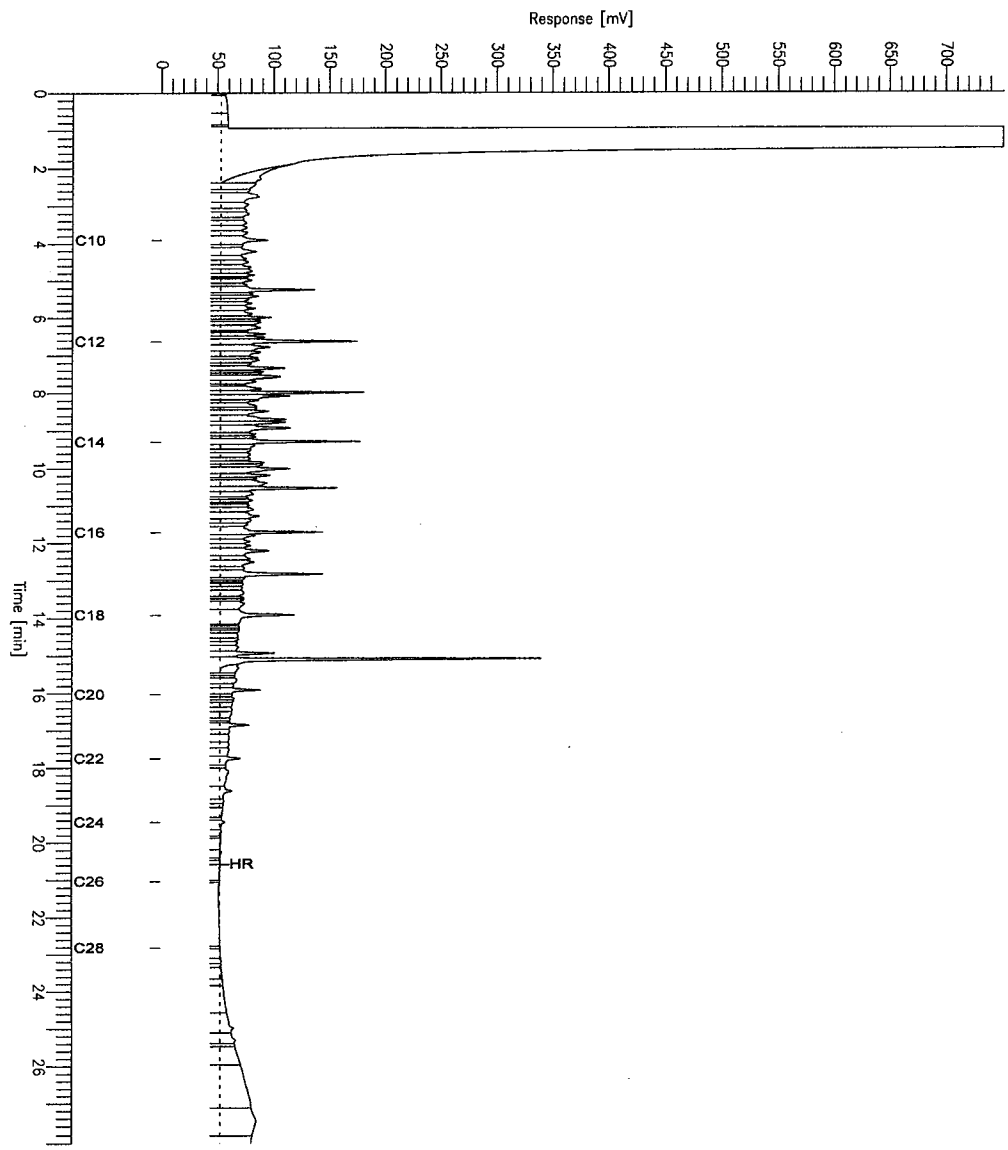
23.3 Standards Log Example

23.4 Analysis Sequence Report Example

23.5 Preparation Batch Report Example

23.6 Method Detection Limit Study Example*
*Only part of the Table or Benchsheet example has been included.

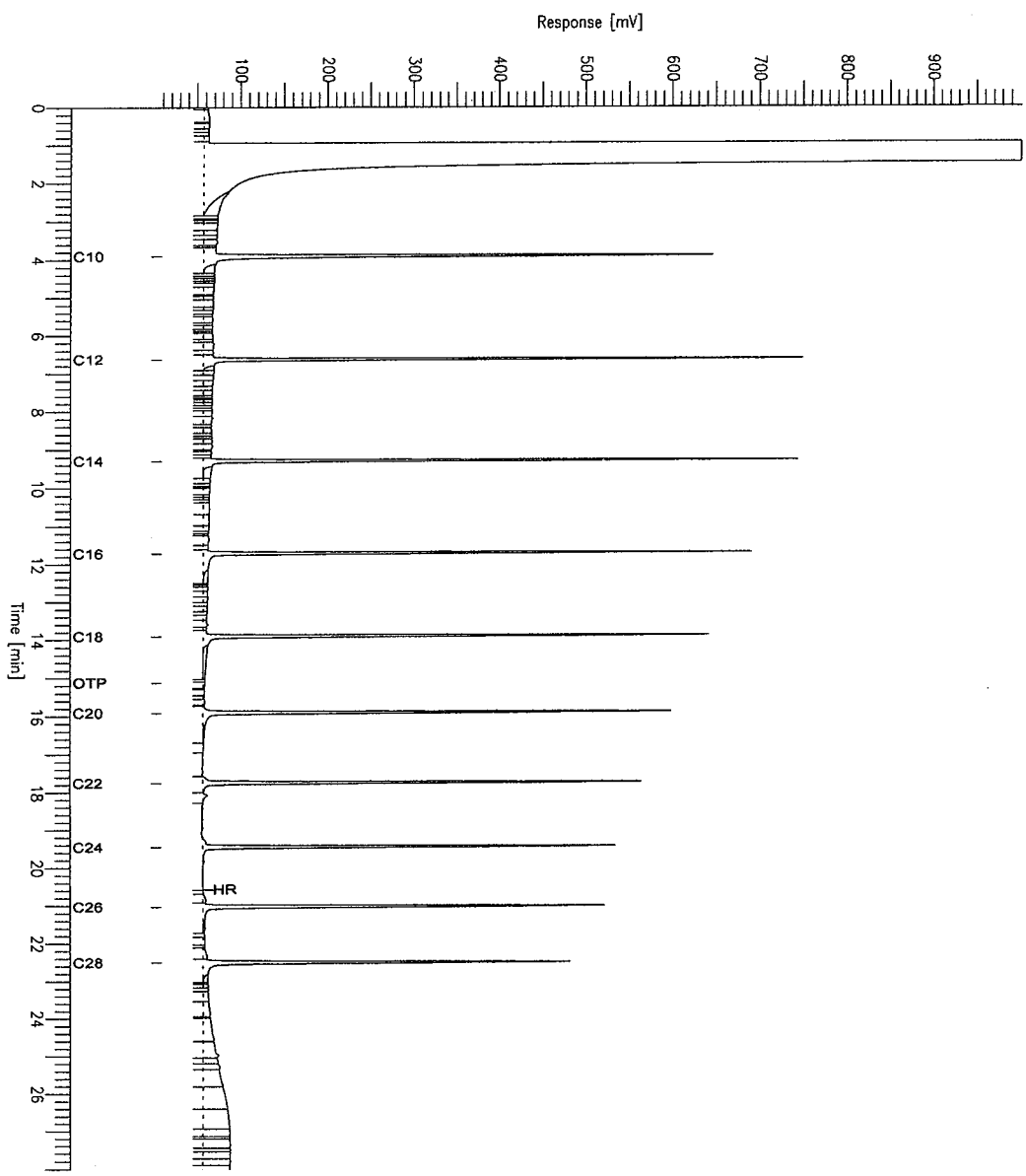
Attachment 23.1
Example Diesel Range Organic Chromatogram on DB-5 Column



Approved By: _____ 1-12-09 **QA Officer**

Approved By: _____ 1/13/9 **Area Supervisor**

Attachment 23.2
Example Retention Time Standard Chromatogram on DB-5 Column



Approved By: _____ 1-12-09 **QA Officer**

Approved By: _____ 1/13/09 **Area Supervisor**

Attachment 23.3
Standards Log Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

8080409

Description:	(AMP) Diesel Fuel #2 2o	Expires:	Aug-19-09
Standard Type:	Other	Prepared:	Aug-13-08
Solvent:	Solvent Lot #A059536	Prepared By:	** Vendor **
Final Volume (mls):	1	Department:	Semivolatiles GC
Vials:	1	Last Edit:	Sep-10-08 16:24 by JMK

Restek part#31259
Lot: A059536
Received 8/13/08

Analyte	CAS Number	Concentration	Units
DRO - 8015B (C10-C35)		50000	ug/mL
DRO - 8015B (C10-C28)		50000	ug/mL

Approved By: MD 1-12-09 **QA Officer**

Approved By: [signature] 1319 **Area Supervisor**

Attachment 23.4
Analysis Sequence Report Example

TriMatrix Laboratories, Inc. **ANALYSIS SEQUENCE** **8122329** Page 1 of 1 Printed: 1/12/2009 5:02:55PM

Semivolatiles GC, Waste, Dec-22-08

Instrument = 157, Calibration = UNASSIGNED

Sequence Analyses:
DRO EPA 8015B

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID*	*Client / QC Type*	*Extraction Comments*
0812259-01	DRO EPA 8015B	A 02			[redacted]	
0814664-BLK1	QC				BLANK	
0814664-BS1	QC				LCS	
0814664-MS1	QC				MATRIX SPIKE	
0814664-MSD1	QC				MATRIX SPIKE DUP	

Approved By: _____ 1-12-09 **QA Officer**

Approved By: _____ 1/13/09 **Area Supervisor**

Attachment 23.5
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH **0814664** Page 1 of 1

Printed: 1/12/2009 4:59:25PM

Semivolatiles GC, Waste, 3580A Waste Dilution

Surrogate #1 = 8070950 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0812259	DRO EPA 8015B				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (g)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client : QC Type*	*Extraction Comments*
0814664-BLK1		Dec-17-08 08:12	BJH	1	10	20				BLANK	
0814664-BS1		Dec-17-08 08:12	BJH	1	10	20		8060406	200	LCS	
0814664-MS1		Dec-17-08 08:12	BJH	1	10	20	0812259-01	8060406	200	MATRIX SPIKE	
0814664-MSD1		Dec-17-08 08:12	BJH	1	10	20	0812259-01	8060406	200	MATRIX SPIKE DUP	
0812259-01	A	Dec-17-08 08:12	BJH	1	10	20				[redacted]	

Approved By: 1-12-09 **QA Officer**

Approved By: 1/13/09 **Area Supervisor**

Attachment 23.6
Method Detection Limit Study Example

SEMI-VOLATILE LABORATORY
INSTRUMENT NUMBER 157 2003 SOIL
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Reference Citation	Date Analyzed	Amount Spiked	Units	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Rep. #6	Rep. #7	Average Amount Found	Average % Recovery	Standard Deviation	MDL
EPA DRO	8015B	1/29/2004	6.67	mg/kg	9.03	9.78	11.9	10.6	10.0	9.79	11.5	10.4	156%	1.03	3.23

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Polychlorinated Biphenyls (PCB) by Gas Chromatography

EPA Method 608
SW-846 Method 8082

APPROVALS:

Area Supervisor: Daniel J. Mierendorf Date: 8/21/09
Daniel J. Mierendorf

QA Officer: Tom C. Boocher Date: 8-21-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 8/21/09
Jeff P. Glaser

Procedure Number: GR-03-128
Revision Number: 2.5

Date Initiated: 4/2/98
Effective Date: 9/5/09

Date Revised: 8/21/09
Pages Revised: All

By: Daniel J. Mierendorf
Total Number of Pages: 43

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

UNCONTROLLED COPY

1.0 SCOPE AND APPLICATION

1.1 This procedure is used to quantitate polychlorinated biphenyls (PCBs) as extracted Aroclors from water, wastewater, soil, wipes, oil, and waste samples. Aroclors are multi-component analytes. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable qualitative and quantitative analysis. The same is true of Aroclors subjected to environmental degradation (weathering), or degradation by treatment technologies. Such degraded mixtures may have significantly different peak patterns compared to Aroclor standards.

1.2 A second analytical technique (different column or GC/MS) must be utilized to confirm positive results.

1.3 Minimum reporting limits (RL) are as follows:

1.3.1 0.100 ug/L for water and wastewater samples

1.3.2 0.017 mg/kg for soil samples

1.3.3 0.500 µg for wipe samples

1.3.4 0.500 mg/kg for diluted waste samples

Reporting limits are highly matrix dependent and the minimum reporting limit might not be achievable with all matrices. Refer to Attachment 21.1 for a default reporting limit list by analyte.

1.4 The linear calibration range begins at the reporting limit and increases to approximately 32 times the reporting limit. The range of analysis may be extended upward by diluting extracts into the linear calibration range.

1.5 Analysis of PCB analytes is restricted for use by or under the supervision of analysts experienced in gas chromatography and chromatographic interpretation.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, December 1996, Method 8082, "Polychlorinated Biphenyls (PCBs) by Gas Chromatography", Revision 0, December, 1996

2.2 *Code of Federal Regulations*, Title 40 Protection of Environment, Volume 19, July 1, 2001 Edition, Chapter I Environmental Protection Agency, Part 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A, Method 608, Organochlorine Pesticides and PCBs

3.0 SUMMARY OF PROCEDURE

3.1 For the analysis, extracts are injected into a gas chromatograph (GC) with two simultaneously loaded columns. The volume injected is 1 or 2 µL.

UNCONTROLLED COPY

3.2 After separation by the column, analyte response is obtained with an electron capture detector (ECD).

3.3 Sample concentration is determined by comparing sample response to a previously run and verified calibration obtained from certified standards of known concentration.

4.0 PARAMETER OR COMPOUND LIST

4.1 Aroclors available for analysis are listed below. Other Aroclors may be analyzed provided an acceptable demonstration of capability is performed which includes all steps of the extraction, cleanup and analysis.

Compound	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

5.0 REFERENCED SOPs

5.1 TriMatrix Laboratories SOP GR-04-101, *Semi-Volatile Organic Laboratory Corrective Actions*, latest revision

5.2 TriMatrix Laboratories SOP GR-09-105, *Waste Dilution Preparation Method for PCBs and Pesticides*, latest revision

5.3 TriMatrix Laboratories SOP GR-09-107, *Extraction of Organochlorine Pesticides and PCBs in Water*, latest revision

5.4 TriMatrix Laboratories SOP GR-09-108, *Extraction of PCBs/Pesticides in Soil, Sludge, and Wastes*, latest revision

5.5 TriMatrix Laboratories SOP GR-09-109, *Sulfur Cleanup*, latest revision

5.6 TriMatrix Laboratories SOP GR-09-110, *Sulfuric Acid Cleanup*, latest revision

5.7 TriMatrix Laboratories SOP GR-09-111, *Florisil Column Cleanup*, latest revision

5.8 TriMatrix Laboratories SOP GR-09-120, *Florisil/Silica Gel cleanup of PCB's/Toxaphene/Chlordane*, latest revision

UNCONTROLLED COPY

5.9 TriMatrix Laboratories SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.10 TriMatrix Laboratories SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Samples often have components that interfere with electron capture detection and/or capillary column chromatography. To minimize interference, extracts are cleaned up before analysis.

6.2 Solvents can introduce oxygenates and sulfur compounds when concentrated after sample extraction giving an undesirable ECD response. Such interferences often coelute with PCB congeners. All extraction solvents must be demonstrated interference-free by the analysis of blanks. Use only pesticide grade or better solvents. Each solvent lot from a vendor must be tested for acceptance prior to use. Once the lot is documented to be contaminant-free by the analysis of at least one extraction blank that indicates no peaks, a large quantity of the test lot is sequestered from the vendor to ensure consistent and verified purity.

6.3 Other sources of contamination are extraction equipment. Rinse all washed glassware with pesticide grade or better methylene chloride immediately before use. Use only high reagent grade or better reagents.

6.3 Carryover can occur whenever highly concentrated samples precede the analysis of low or non-detect samples. To reduce carryover, autosampler syringes are rinsed between each injection. When necessary, analyze a solvent blank immediately after a high level sample. If carryover is suspected and/or observed in the solvent blank, all affected samples must be re-analyzed.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 PCBs can cause irritation upon contact and are carcinogenic. Open, prepare and dispense standards and solutions only in a fume hood. Wear a laboratory coat, disposable gloves and safety glasses when working in the laboratory.

7.3 Extraction solvents are toxic. Avoid contact with the skin, avoid inhalation of solvent vapors and do not ingest (no chewing gum in the laboratory). Open solvent containers under a fume hood.

7.4 All analyzed sample extracts and expired standards must be disposed of properly. Consolidate all samples and extracts testing positive for any concentration of PCBs as PCB-containing waste.

7.5 If a PCB or solvent spill occurs, notify the area supervisor and safety officer. Evacuate the area and begin cleaning up the spill as outlined in the safety manual.

7.6 Every six months, an ECD wipe test must be performed on each detector as dictated by federal law. The test is used to determine whether an ECD is leaking radioactive material. Three areas must be checked as follows:

UNCONTROLLED COPY

7.6.1 The detector housing

7.6.2 The detector entrance

7.6.3 The detector exhaust

7.6.4 Wipe tests are sent to the National Leak Test Center for testing. Leak test reports are kept on file in the safety office.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Water, wastewater and leachate samples are collected in unpreserved 1000 mL amber glass jars with PTFE septum-lined lids then stored at 0 – 6° C until extraction. At least two 1000 mL aliquots of aqueous sample must be collected to meet reporting limit requirements or to re-extract when necessary. If matrix spikes are needed, collect two additional 1000 mL sample volumes.

8.2 Waste, sludge and soil samples are collected in glass jars with no preservative added. At least 30 g of solid is needed to meet reporting limit requirements. Additional sample must be collected to re-extract when necessary, for matrix spikes and for a percent solids determination.

8.3 Clean wipes are sent to the client in 125 mL glass jars with PTFE septum-lined lids, with 30 mL of hexane in the jar.

8.4 The water sample extraction must be performed within 7 days of the date of collection for method 8082 and within 365 days for method 608. Soils must be extracted within 14 days of the date of collection. Extract analysis must be completed within 40 days of sample extraction for method 8082 and within 365 days for method 608.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Gas chromatograph equipped with dual $_{63}$Ni electron capture detectors and flow valves capable of accurately delivering very low flow rates (~10 mL/min). Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

9.1.1 Instrument Set-up:

9.1.1.1 Injector Temperature: 205° C isothermal

9.1.1.2 Detector Temperature: 350° C

9.1.1.3 Oven Program: Initial temperature at 120° C (no hold), ramp to 160° C at 20° C/min (no hold), ramp to 300° C at 9° C/min, hold 3.67 minutes

9.1.1.4 Carrier Flow: 6.0 mL/min

9.1.2 Autosampler: Leap A200SE or Combi-Pal™ programmable injectors

UNCONTROLLED COPY

9.2 Software operated on a personal computer workstation possessing the capability to collect and archive analysis data. Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer specifications associated with the analytical instrument.

9.3 Columns:

9.3.1 Column A: RTX CLP, 30 m length, 0.53 mm ID, 0.50 µm film thickness.

9.3.2 Column B: RTX CLP2, 30 m length, 0.53 mm ID, 0.42 µm film thickness.

9.4 Microsyringes, glass, various sizes, gas-tight

9.5 Autosampler vials with PTFE-faced silicone septa, 2.0 mL

9.6 Amber vials, with PTFE-lined screw caps, 40 mL

9.7 Volumetric flasks, 10, 100, and 1000 mL class A

9.8 Analytical balance, capable of accurately weighing to 0.0001 g

9.9 Pasteur Pipets, 2 mL, disposable, glass

9.10 Vials, borosilicate glass with PTFE-lined screw caps, 10 mL

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Direct inject gas chromatographs are equipped with silicone septa (Supelco Thermogreen or equivalent) which eventually core and leak. These must be replaced to avoid contaminating the injection port liner with rubber particles and to eliminate column bleed. Replace septa frequently enough to prevent leaking and bleed problems and/or after approximately 100 injections.

10.2 Injection port liners can become a source of contamination and flow restriction by accumulating sample debris and septum particles. Periodically replace the injection port liners to minimize the loss of peak resolution.

10.3 When conditions merit, clip about 6" off the front of the analytical column. When column resolution or chromatography deteriorates and cannot be solved by column clipping, the column must be replaced.

10.4 Electron capture detectors may begin to bleed radioactive particles as they age. Wipe tests must be conducted every six months to ensure there is no bleed. The cause is that sample components and constant use at high temperature oxidize the $_{63}$Ni foil inside a detector. Foil degradation also raises baseline noise and decreases the linear calibration range. When this happens, send the detector out for cleaning and/or refoiling. NEVER tamper with any internal ECD part as this could lead to radiation exposure. All ECD repair must be done by an outside licensed professional.

10.5 Replace gas filters annually.

UNCONTROLLED COPY

11.0 CHEMICALS AND REAGENTS

11.1 PCB standards are purchased as certified solutions in heat-sealed ampules.

11.2 Hexane, pesticide grade or better

11.3 Helium carrier gas, ultra high purity

11.4 Nitrogen makeup gas, filtered for oxygen, hydrocarbons and moisture

11.5 Surrogate standards – tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB), purchased as certified solutions.

12.0 STANDARDS PREPARATION

12.1 Stock Standards

12.1.1 Aroclor stock standards are purchased as certified solutions in hexane or methanol at 1000 ug/mL which are serially diluted in hexane. Stock standards are stored in 100 mL amber screw-cap bottles at 0 – 6° C and away from light. Being multicomponent analytes, PCB standards are prepared independently except for 1221/1254 and 1016/1260. These analyte pairs are combined due to dissimilar elution times.

12.1.2 Surrogate stock standards are purchased as certified solutions in acetone, typically at 200 ug/mL.

12.1.2 All stock standards must be replaced after one year or sooner if comparison with calibration standards indicates a problem.

12.2 Intermediate standards are made by diluting stock standards in hexane (1:100). Intermediate standards are stored in 40 mL amber screw-cap vials at 0 – 6° C and away from light.

12.3 Calibration Standards

12.3.1 A standard of Aroclors 1016/1260 includes the majority of peaks represented in the other five Aroclors. Consequently, a multi-point initial calibration of the 1016/1260 mixture at six concentrations demonstrates detector response linearity without performing independent calibrations for each of the seven Aroclors.

12.3.2 Prepare six calibration standards containing equal concentrations of Aroclors 1016 and 1260, and equal concentrations of surrogates TCMX and DCB. Dilute with hexane. The following concentrations correspond to the expected sample concentration range and bracket the linear detector response: 0.04, 0.08, 0.16, 0.32, 0.64 and 1.28 ug/mL for 1016/1260, and 0.005, 0.01, 0.02, 0.04, 0.08, 0.16 ug/mL for TCMX/DCB.

UNCONTROLLED COPY

12.3.3 The lowest calibration standard is the minimum reporting limit.

12.3.4 The same calibration concentrations are used if a multi-point calibration is used for Aroclors other than 1016/1260. However, an acceptable linear multi-point 1016/1260 calibration justifies the use of a single-point calibration for quantitation of other Aroclors except when otherwise specified.

12.3.5 Standards of each Aroclor are required for pattern recognition. Assuming linearity has been demonstrated by the 1016/1260 calibration, a single-point calibration may be used for pattern recognition. Prepare a standard at 0.32 ug/mL for each Aroclor and 0.04 ug/mL for TCMX/DCB.

12.4 All standards must be labeled with the following information:

12.4.1 Initials of preparer

12.4.2 Preparation date

12.4.3 Concentration, with units

12.4.4 Standard name

12.4.5 Standard identification number as it appears in the standards log

12.4.6 Expiration dates must be no later than six months from the date of preparation for intermediate and working standards and no later than one year for purchased stock standards that have been opened and stored without headspace. The expiration date of an intermediate or working standard cannot exceed the purchased standard(s) expiration date from which it was made.

12.4.7 Solvent used

12.5 All standards preparation must be recorded in the standards log and uniquely identified. Refer to Attachment 21.2 for an example standards log.

13.0 SAMPLE PREPARATION

13.1 Water samples are extracted in accordance with TriMatrix SOP GR-09-107.

13.2 Soil and wipe samples are extracted in accordance with TriMatrix SOP GR-09-108

13.3 Waste dilution samples are extracted in accordance with TriMatrix SOP GR-09-105.

13.4 Extracts are cleaned up for sulfur in accordance with TriMatrix GR-09-109.

13.5 Extracts are cleaned up with sulfuric acid in accordance TriMatrix GR-09-110.

UNCONTROLLED COPY

13.6 Extracts are cleaned up with Florisil and silica gel in accordance with TriMatrix SOP GR-09-111 or TriMatrix SOP GR-09-120.

14.0 CALIBRATION PROCEDURES

14.1 Initial Calibration

14.1.1 Following a clean hexane blank, run a 6-point curve for Aroclors 1016/1260 (refer to Section 12.3.2 for concentrations). The low standard, when extracted, is equivalent to the minimum reporting limit (500 ug/L for waste dilution, 0.1 ug/L for water, ~17 ug/kg for soil and 500 ug/wipe for wipes). A single-point 0.32 ug/mL calibration standard is used for pattern recognition of other Aroclors.

14.1.1.1 Inject 1.0 µL of each calibration standard. Choose five peaks for quantitation, which are characteristic of the pattern. Record the height of each Aroclor peak chosen. Try to choose peaks that are at least 25% of the height of the largest peak in the Aroclor pattern. Later-eluting peaks are generally more stable for quantitation.

14.1.2 Determine the average calibration factor (CF_{avg}) for the sum of the chosen peaks using the following equations:

$$\text{Calibration Factor (CF)} = \frac{\text{sum of the chosen peak responses}}{\text{Aroclor concentration, ug/mL}}$$

$$\text{Average Calibration Factor } (CF_{avg}) = \frac{\text{sum of calibration factors } (CF_{sum})}{\text{number of calibration points}}$$

14.1.3 Calculate the relative standard deviation (%RSD) of CF_{avg} using the following equation:

$$\text{percent relative standard deviation (\%RSD)} = \frac{\text{standard deviation of } CF_{avg}}{CF_{avg}} \left| 100 \right.$$

Note: The RSD value must be ≤20% to assume the calibration passes through the origin to use average calibration factor when reporting by method 8082 (or ≤10% for method 608). If the calibration does not in fact pass through the origin, do not use CF_{avg} even if %RSD is acceptable.

14.1.4 If %RSD is greater than the method specified criterion (10% for 608 and 20% for 8082), perform instrument maintenance and run the calibration again. Polynomial regression must not be used if the calibration factor has previously been acceptable. Instrument maintenance must be performed to obtain an acceptable average calibration factor or linear regression. When linear regression is used, the correlation coefficient (r) must be ≥0.99.

UNCONTROLLED COPY

Note: If %RSD is >20%, the calibration does not actually pass through the origin, or if linear regression is used, a multi-point regression must be used for quantitation where a single-point calibration CF_{avg} of Aroclor 1221, 1232, 1242, 1248, 1254, 1262 or 1268 might otherwise be allowed.

Note: Single-point calibration may be used to report by method 8082 only if the project and/or data quality objective does not require a multi-point calibration.

Note: For samples analyzed by method 608 or if project and/or data quality objectives dictate, detected Aroclors other than 1016/1260 must be re-analyzed with a multi-point calibration of the Aroclor.

14.1.5 Construct linear regression curves by plotting peak response (height) against concentration. The regression must not be forced through the origin:

14.1.5.1 Linear regression requires five calibration points and is calculated as follows:

$C_s = (H_s - b)/a$

Where:
H_s = Height of the target analyte peak, in sample "s"
C_s = Concentration of the target analyte in calibration standard "s"
a = Slope "s" of the curve (also called the coefficient of C)
b = The intercept

14.1.5.2 Do not select regression as a calibration option unless it has been proven that after instrument and column maintenance, calibration does not characteristically go through the origin and/or has not previously generated a consistently acceptable average calibration factor.

14.1.6 Sample analysis may not begin without an acceptable 1016/1260 calibration. For an Aroclor 1260 calibration curve example, refer to Attachment 21.3.

14.1.7 Initial calibration if analyzing by method 608 is as follows:

14.1.7.1 Initial calibration consists of a minimum of three concentration levels for all Aroclors.

14.1.7.2 The %RSD must be <10% to use an average calibration factor for quantitation.

14.1.7.3 If %RSD is >10%, a minimum of a three-point linear calibration curve must be constructed for ALL Aroclor quantitation, not just 1016/1260. The correlation coefficient (r) must be ≥0.99 for an acceptable regression.

14.2 Second-Source Calibration Verification (SCV)

UNCONTROLLED COPY

14.2.1 Prepare a second-source calibration verification standard (SCV) at 0.40 ug/mL to validate the initial calibration Aroclors 1016/1260 solutions. Acceptance limits for the SCV are listed in the laboratory information management system (Element™)

14.2.2 If recovery is unacceptable, re-run the SCV once. If still unacceptable, re-prepare the SCV and re-analyze successfully. If the second SCV fails, re-calibrate and re-analyze the SCV successfully. Sample analysis may not begin without an acceptable SCV being run.

14.3 Continuing Calibration Verification (CCV)

14.3.1 On non-curve days, analyze a mid-point CCV containing 0.32 ug/mL of Aroclors 1016/1260 and surrogates (0.05 ug/mL). Calculate percent difference or percent drift. Regardless of calibration technique, percent difference or percent drift must be within ±15% of the expected concentration to be acceptable.

14.3.2 When using average calibration factor, verification is performed using percent difference:

$$Percent\ Difference = \frac{CF_{ccv} - CF_{avg}}{CF_{avg}} \times 100$$

where:
CF_{ccv} = Response factor from verification standard
CF_{avg} = Average calibration factor from initial calibration

14.3.3 When using linear regression, verification is performed using percent drift:

$$\text{Percent Drift} = \frac{R_2 - R_1}{R_1} \times 100$$

where:
R_1 = Expected concentration, in ug/mL
R_2 = Observed concentration, in ug/mL

14.4.3 A CCV is not required on the remaining Aroclors unless a multi-point regression is used.

14.4.4 Quantitate based on the following criteria:

14.4.4.1 If an Aroclor other than 1016/1260 is identified using method 8082, quantitate using the single-point standard run with the initial 1016/1260 calibration.

Note: Single-point calibration may be used for method 8082 samples only if the 1016/1260 calibration model is CF_{avg} but may not be used if project and/or data quality objectives specify otherwise.

14.4.4.2 For Aroclors 1016 or 1260, use the initial 1016/1260 calibration.

UNCONTROLLED COPY

15.0 ANALYTICAL PROCEDURE

15.1 The analysis sequence begins with a hexane blank (BLK), followed by an initial calibration or on non-curve days by the 0.32 ug/mL Aroclor 1016/1260 CCV. Analyze the other Aroclor CCVs, at 0.32 ug/mL, at this time for confirmation analysis.

15.2 After every 10 injections, the 1016/1260 CCV must be analyzed to verify the calibration is in control. Both Aroclors in the CCV must have less than ±15 percent difference or drift from the initial calibration. If any CCV Aroclor fails the ±15 percent criteria on the low side (less than the expected value), all extracts run before and after the failing CCV must be re-injected on a calibration that is in control. If any CCV Aroclor fails the ±15 percent criteria on the high side (greater than the expected value), only those extracts with detectable results need re-injected.

Note: At least once per month, vary the CCV concentration to demonstrate detector and calibration accuracy at concentrations other than the mid-point.

15.3 Identify individual PCB congeners by comparing the congener retention time against the initial calibration peak. For positive identification, the peak retention time must elute within the calculated retention time window on both columns. However, PCB Aroclors must be identified by pattern recognition

15.3.1 Many Aroclors contain identical congeners. However the relative peak height is unique for each, with each Aroclor having a distinct pattern. To identify and report an Aroclor, the sample pattern of peaks must match the standard. Often, a pattern may be difficult to isolate because of a complex matrix or weathering. Consequently, experience and analytical judgment must be used in the identification process. Aroclor 1221 is unique and presents a challenge. It elutes early and has a simple pattern. All 1221 congeners are part of other Aroclors and are most affected by matrix. Exhibit caution in the identification of Aroclor 1221.

15.3.2 Aroclors can be grouped into two groups. Early eluting Aroclors (Attachment 21.9 – 21.18) are as follows:

15.3.2.1 Aroclors 1221, 1232, 1242, 1248 and 1016 are early eluters. The initial and most significant criterion for identification is the proportional relationship between peaks 1 through 7 in Attachment 21.8 (key identification peaks). Secondly, the absence or presence of peaks 8 through 14.

15.3.2.2 Quantitation

Use at least three peaks, typically five from 1 through 7 (Attachment 21.8) except for 1221 (Attachment 21.12). Do not use disproportionate peaks to avoid matrix interference. As a rule of thumb, relative peak ratios used for quantitation in samples must be within 25% of the ratios in the identified Aroclor standard.

Note: If there is an obvious overlapping peak or other matrix interference precluding the use of any chosen calibration peak, recalibrate and report using the sum of the unaffected peaks and discuss in the report narrative that matrix interference precluded the use of all chosen calibration peaks.

UNCONTROLLED COPY

15.3.3 Late eluting Aroclors (Attachments 21.9 – 21.12, 21.19 – 22.22)

15.3.3.1 Late eluting Aroclors are usually easier to identify. The following criteria apply:

15.3.3.1.1 Aroclors 1254, 1260, 1262, and 1268 are late eluters

15.3.3.1.2 The second indication is the absence or presence of peaks 8 through 14 (Attachment 21.8).

15.3.3.1.3 The third indication is the relationship of peaks 8 through 13 to each other.

15.3.3.1.4 The fourth indication is the proportion of later eluting peaks.

15.3.3.2 Quantitation. Use at least three congener peaks, preferably five. Use peaks later than peak 7 (Attachment 21.8) if early Aroclors are present. Do not use disproportionate peaks, to avoid matrix interference.

15.3.4 Samples containing more than one Aroclor.

15.3.4.1 Once an Aroclor is identified, the same peaks cannot be used to identify a different Aroclor since it is impossible to identify 1248 and 1242 in the same sample. If both are present it will appear to be one or the other. Early and late eluting Aroclors are not clearly divided. If an Aroclor is present from each elution group, patterns may be distorted by overlapping peaks. This is important when attempting to identify multiple Aroclors. High levels of an early eluting Aroclor may appear to be later eluting peaks of a separate Aroclor and care must be taken to look at relative peak intensities when comparing with standard patterns.

15.3.4.2 Peaks 1 through 7 must be used for quantitating early eluting Aroclors (Attachment 21.8). Peaks 8 through 14 must be used for late eluting Aroclors. A minimum of three peaks (preferably five) from each elution group are required for quantitation. These peaks have been shown to be at least 25% of the highest peak. A calibration must be performed if using other congeners than those chosen for the initial calibration, with samples and all quality control quantitated against the new calibration.

15.4 Retention Time Windows

15.4.1 Perform a retention time study by making three injections of each Aroclor (not just the 1016/1260 mixture) over a 72-hour period and average the results. Calculate the mean and standard deviation for each peak used in quantitation. Calculate the retention time window by multiplying the standard deviation by ±3.

15.4.2 For a positive identification based on retention time, all congener quantitation peaks must elute within the absolute retention time.

UNCONTROLLED COPY

15.4.3 Since an initial CCV is run before sample analysis each day/shift, establish the center of the retention time window for the day/shift using the initial CCV quantitation peak retention times. Establish the retention time window using the initial CCV retention times plus or minus three times the retention time study standard deviation.

15.4.4 All Aroclor peaks chosen for quantitation in samples, quality control and standards run after the ICV must elute within the retention time window. Samples run in association with any subsequent CCV eluting outside the retention time window must be re-analyzed.

15.4.5 Update retention time windows whenever a new column is installed.

15.5 Example chromatograms showing Aroclor patterns on the RTX-CLP and RTX-CLP2 columns are provided in Attachments 21.9 – 21.22.

15.6 All targeted analyte identifications must be confirmed by running on the secondary column or by gas chromatography/mass spectrometry (GCMS).

15.6.1 When confirmation is made using a second column, the secondary analysis must meet all QC criteria described for the primary column, including calibration and retention times. Evaluate the agreement between results after identification has been confirmed by relative percent difference.

15.6.2 Calculate relative percent difference (RPD) using the formula below where R_1 and R_2 are the two results. The vertical bars in the numerator indicate the absolute value of the difference. Therefore, RPD is always a positive value.

$$RPD = \frac{|R_1 - R_2| \, 200}{R_1 + R_2}$$

15.6.3 If one result is significantly higher (if RPD is greater than 40%), check to see if an obviously overlapping peak is causing one erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. If no anomalies are noted, review the chromatographic conditions.

15.6.4 If there is no evidence of chromatographic problems such as overlapping peaks, report the lower result (unless otherwise specified) with narration as to the disparity between column results.

15.6.5 If RPD is less than 40%, report from the primary column.

15.7 If an extract concentration exceeds the calibration range, dilute and re-analyze. All extract dilutions must keep average peak response in the upper half of the calibration range. Re-dilute if concentrations are outside this range.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Aroclors identified in samples must be quantitated by comparison with corresponding standards.

UNCONTROLLED COPY

16.2 Once an Aroclor has been identified, the sum of the height response of congener peaks are compared to the sum of height response of corresponding peaks in the calibration in accordance with Section 14.0.

16.3 If any chosen quantitation peak needs omitted due to interference, a minimum of three peaks must remain for quantitation. The calibration must be adjusted by removing any rejected congener peak from the summed peak response. Quality control must also be re-quantitated using the new summed peak response and acceptance re-verified.

16.4 Sample concentration is calculated using the extract Aroclor concentration in one of the following equations:

Water Samples:

$$\text{Sample Concentration (in ug/L)} = \frac{(\text{ug/mL})(V_e)(DF)}{(V_w)}$$

Wipes:

$$\textit{Wipe Concentration (in total ug)} = (ug/mL)(Ve)(DF)$$

Soil Samples:

$$\text{Sample Concentration (in mg/kg)} = \frac{(\text{ug/mL})(\text{Ve})(\text{DF})}{(\text{Vs})(\text{PS})} \times 1000$$

where:

V_e = Total volume of extract, in µL
DF = Post extraction dilution factor as a ratio
V_w = Initial water sample volume, in mL
V_s = Initial soil sample mass, in g
PS = Soil percent solids in decimal form (0.98 not 98%)

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for data quality and for filling in all documentation and paperwork correctly. It is important to document analysis by correctly filling in, handing in and archiving all paperwork correctly.

17.2 Laboratory Information Management System (Element™) data transfer must be in accordance with TriMatrix SOP GR-10-123. If internal chain-of-custody is required, it is very important that a CoC form be filled in correctly and completely.

17.3 All run, maintenance, CD archival and standard logs must be filled in completely and correctly. Corrections on hardcopy must be made with a lineout (not a writeover) and must be initialed and dated. Blank lines in run logbooks must be Z'd out. Refer to Attachment 21.6 for a run logbook example.

17.4 All calibration run hardcopy must be archived in the correct storage box.

UNCONTROLLED COPY

17.5 All other analysis documentation must be archived appropriately for ease in retrieval.

18.0 QUALITY ASSURANCE

18.1 Blanks, surrogates and matrix spikes must be included as part of quality control.

18.2 Before calibration, initial CCV or samples are run, a solvent blank must be analyzed to demonstrate the analytical system free from interferences and contamination at the reporting limit. Solvent blanks must be run more frequently when showing evidence of carryover. The extraction blank (BLK) may be used as a solvent blank to minimize runs.

18.2.1 The extraction blank (BLK) is laboratory reagent water extracted with hexane as a sample and containing surrogates.

18.2.2 The BLK must be non-detect at the minimum reporting limit.

18.2.3 At a minimum, the BLK is run at the beginning of every analytical shift and/or with each batch of up to 20 samples. Whichever is more frequent.

18.2.4 The BLK must be extracted more frequently if showing contamination to isolate the problem.

18.2.5 Extraction blanks must be prepared as samples, in accordance with all extraction procedure requirements, including cleanup.

18.2.6 Refer to TriMatrix SOP GR-04-101 for corrective action during blank contamination.

18.3 Surrogate criteria are as follows:

18.3.1 All samples must be spiked with tetrachloro-m-xylene and decachlorobiphenyl surrogates. Until twenty samples of a given matrix have been analyzed, acceptance limits will be 50-150% recovery. Once twenty samples of a given matrix have been analyzed, statistical acceptance limits must be generated in Element™ and used. At a minimum, surrogate recovery limits will be updated annually on a matrix-by-matrix basis. Calculate surrogate recovery for each extract analysis. If recovery or precision is not within acceptable limits, consult TriMatrix SOP GR-04-101 to determine when and how data is qualified.

18.3.1.1 High surrogate recovery may be due to co-eluting matrix interference from the sample. Examine the chromatogram for evidence of co-elution. No corrective action is required in this instance.

18.3.1.2 Low recovery may be due to poor extraction efficiency in the matrix. Verify by re-extracting the sample if sufficient sample volume and hold time permits.

18.3.1.3 If surrogate recovery in a BLK is below the lower control limit (LCL), only samples with failing surrogate recovery require re-extraction if sufficient sample volume and hold time permits. If BLK surrogate recovery is above the upper

UNCONTROLLED COPY

control limit (UCL), no corrective action is required if sample surrogate recovery is acceptable.

18.3.1.4 If surrogate recovery is outside control limits in the matrix spike and/or matrix spike duplicate, re-analysis is only required if blank spike (BS) recovery is also outside control limits. If the BS is out-of-control after re-analysis, all associated samples must be re-extracted if sufficient sample volume and hold time permits.

18.3.2 Control limits are calculated as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

where:
p = average percent recovery
s = standard deviation of the percent recovery

18.3.3 If surrogate recovery is not within control limits, take corrective action based on TriMatrix SOP GR-04-101, which outlines sample re-extraction if sufficient sample volume and hold time permits and/or data qualification. If sample volumes and holding times permit, samples with low recoveries must be re-extracted, unless matrix interference indicates otherwise. Samples with high recoveries must be re-extracted only if there are detectable results. If many samples are out-of-control for no apparent reason, the gas chromatograph must be inspected for mechanical failure and appropriate repairs made. Once repairs have been made, extracts run in association with the failure must be re-analyzed.

18.3.4 Continuing calibration verification (CCV) surrogate recovery is handled as follows:

18.3.4.1 If a CCV has a surrogate recovery outside control limits but surrogate recovery in all samples is in control, instrument recalibration and extract re-analysis is not required.

18.3.4.2 If the CCV and sample extracts have high surrogate recovery, sample extracts must be rerun after instrument recalibration.

18.3.4.3 If the CCV and sample extracts have low recovery, sample extracts must be rerun after instrument recalibration.

18.4 Matrix Spikes (MS) and Blank Spikes (BS)

18.4.1 Extract a matrix spike and matrix spike duplicate at a frequency of once for each batch of up to 20 samples for each matrix.

18.4.1.1 If one to ten samples are extracted in a month, at least one matrix spike and one matrix spike duplicate must be extracted.

18.4.1.2 When reporting by method 608, matrix spike at a frequency of once for each batch of up to 10 samples.

UNCONTROLLED COPY

18.4.1.3 When reporting by method 608, extract the blank spike once for each batch of up to 10 samples. Otherwise, a blank spike must be extracted every shift samples are extracted, regardless of the number of samples extracted.

18.4.1.4 If there is insufficient sample volume to perform matrix spikes, extract a duplicate blank spike.

18.4.2 Until 15-20 matrix spikes, matrix spike duplicates and blank spikes have been analyzed, evaluate recovery against default limits of 50-150% and a duplication limit of 40%. Once generated, statistical control limits are listed in Element™.

18.4.3 Control limits must be updated at least annually.

18.4.4 Calculate matrix spike recovery is calculated as follows:

$$\text{Percent Recovery} = \frac{(\text{Aspk} - \text{Asmp})}{\text{SPK}} \times 100$$

where:
Aspk = Concentration in the spiked sample (ug/L)
Asmp = Concentration in the unspiked sample (ug/L)
SPK = Concentration spiked (ug/L) [(concentration of spike standard in ug/mL x mL spiked) / initial sample volume]

18.4.5 Calculate relative percent difference (RPD) as follows:

$$RPD = \frac{|SPK_1 - SPK_2|}{SPK_1 + SPK_2} 200$$

where:
SPK_1 = Matrix Spike
SPK_2 = Matrix Spike Duplicate

18.4.6 If recovery or duplication is not within acceptable limits, perform corrective action in accordance with TriMatrix SOP GR-04-101.

18.4.7 If a blank spike is out-of-control, the problem must immediately be identified and corrected before further samples are extracted. The purpose of an LFB is to verify that out-of-control analytes in a matrix spike are the result of matrix interference rather than extraction or instrumental error. Failure of any part of a blank spike requires corrective action up to and including re-extraction of all associated samples. The only exception is when blank spike recovery is high and all associated samples are below the reporting limit.

18.4.8 Every effort must be made to determine the reason for a blank spike failure (whether mis-spiked, mis-extracted or something else) and appropriate actions taken. A non-conformance report must be written that outlines the investigation and findings. Corrective action must be in accordance with TriMatrix SOP GR-04-101.

UNCONTROLLED COPY

18.4.9 The Aroclor used for matrix and blank spiking must be changed periodically throughout the year so all analytes are spiked.

19.0 ANALYST CERTIFICATION/METHOD VALIDATION

19.1 Before sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an initial demonstration of capability (IDC) study. While a demonstration of capability study is not instrument-dependent, a study is required on every instrument and every column to demonstrate an acceptable level of accuracy and precision.

19.2 Spike 1.0 mL of 1016/1260 blank spiking solution into each of four, 1 L aliquots of water and extract as samples. Cleanup is not necessary for the study unless the extraction analyst is performing a demonstration of capability study also. The blank spiking solution is not to be a standard used for calibration.

19.3 Analyze the four extracted spikes following every step in this procedure.

19.4 Input results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and standard deviation for each Aroclor.

19.4.1 Average recovery must be within blank spike control limits and standard deviation must be ≤20% RSD. If both criteria pass, the analyst and the instrument/column used are authorized to run samples.

19.4.2 If any criterion fails, the demonstration of capability study must be repeated.

19.5 For a failed study, locate and correct the source of the problem and repeat the study successfully. Repeated failure will indicate a general problem with the procedure and/or techniques used. If this occurs, locate the problem, correct the procedure and/or techniques used and repeat the study successfully.

19.6 Samples may not be analyzed by any analyst or on any instrument until a successful demonstration of capability study has been completed and submitted to the quality control department for approval and documentation.

19.7 A Method Detection Limit (MDL) study must be performed for each Aroclor (not just for 1016/1260). MDL studies must be performed on all instruments involved in PCB analysis, on both columns and for each preparation procedure. Refer to TriMatrix SOP GR-10-125.

20.0 REFERENCES

20.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, December 1996, Method 8082, "Polychlorinated Biphenyls (PCBs) by Gas Chromatography", Revision 0, December, 1996

20.2 *Code of Federal Regulations*, Title 40 Protection of Environment, Volume 19, July 1, 2001 Edition, Chapter I Environmental Protection Agency, Part 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A, Method 608, Organochlorine Pesticides and PCBs

UNCONTROLLED COPY

21.0 ATTACHMENTS/APPENDICES

21.1 Default Reporting Limits

21.2 Standards Log Example

21.3 Sample of Calibration Curves for PCB 1016/1260

21.4 Preparation Batch Report Example

21.5 Analysis Sequence Report Example

21.6 Sample Page of Instrument Run Logbook

21.7 Method Detection Limit Study Example

21.8 Key Peaks for Aroclor Identification

21.9 1016/1260 on RTX CLP Column

21.10 1016/1260 on RTX CLP2 MS Column

21.11 1221/1254 on RTX CLP Column

21.12 1221/1254 on RTX CLP2 Column

21.13 1232 on RTX CLP Column

21.14 1232 on RTX CLP2 Column

21.15 1242 on RTX CLP Column

21.16 1242 on RTX CLP2 Column

21.17 1248 on RTX CLP Column

21.18 1248 on RTX CLP2 Column

21.19 1262 on RTX CLP Column

21.20 1262 on RTX CLP2 Column

21.21 1268 on RTX CLP Column

21.22 1268 on RTX CLP2 Column

UNCONTROLLED COPY

Attachment 21.1
Default Reporting Limits

UNCONTROLLED COPY

Attachment 21.1
Default Reporting Limits

Compound	Water (ug/L)	Soil (mg/kg)
PCB-1016	0.10	0.33
PCB-1221	0.10	0.33
PCB-1232	0.10	0.33
PCB-1242	0.10	0.33
PCB-1248	0.10	0.33
PCB-1254	0.10	0.33
PCB-1260	0.10	0.33
PCB-1262	0.10	0.33
PCB-1268	0.10	0.33

Compound	Wipes (ug)	Waste (mg/kg)
PCB-1016	0.5	0.50
PCB-1221	0.5	0.50
PCB-1232	0.5	0.50
PCB-1242	0.5	0.50
PCB-1248	0.5	0.50
PCB-1254	0.5	0.50
PCB-1260	0.5	0.50
PCB-1262	0.5	0.50
PCB-1268	0.5	0.50

Attachment 21.2
Standards Log Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
A604465

Description:	(AMP) Aroclor 1016	Expires:	Sep-30-08
Standard Type:	Other	Prepared:	May-12-06
Solvent:	Solvent Lot #W-125M-07-02	Prepared By:	** Vendor **
Final Volume (mls):	1	Department:	Semivolatiles GC
Vials:	2	Last Edit:	Apr-12-07 13:29 by JLW

NSI part# 125M
LOT#125M-07-02
Received 10/31/06

Analyte	CAS Number	Concentration	Units
PCB-1016	12674-11-2	1000	ppm
PCB-1016 [2C]	12674-11-2	1000	ppm

Reviewed By _____ Date _____

Page 1 of 1

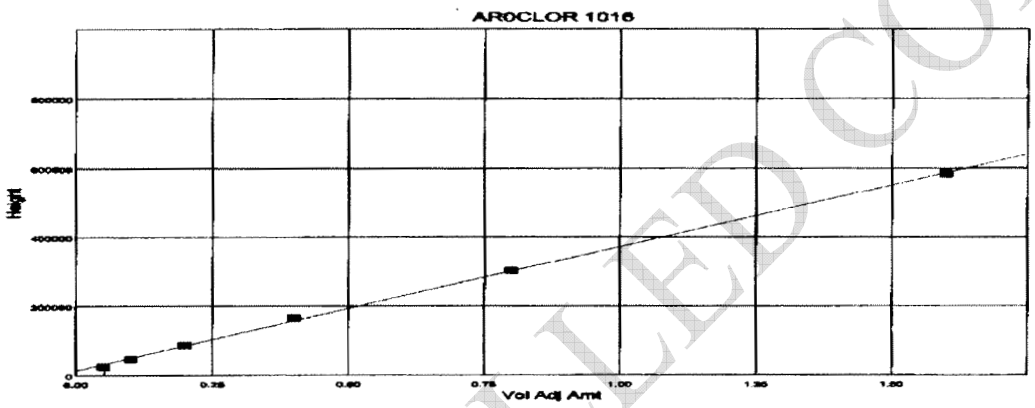
UNCONTROLLED COPY

Attachment 21.3
Sample of a Calibration Curves for PCB 1016/1260

Fit Analysis Output For Method File: "C:\TC4\GC144\AB183\PCBAA.MTH"
Component Name: "AROCLOR 1016"
Date: 11/2/07 Time: 14:17

Curve Parameters:

Curve #1 : 1st Order
Weighting Factor = 1.0 (No Weighting) r^2 = 0.999340
Calibration Curve = (14080.796680) + (358052.114081)X

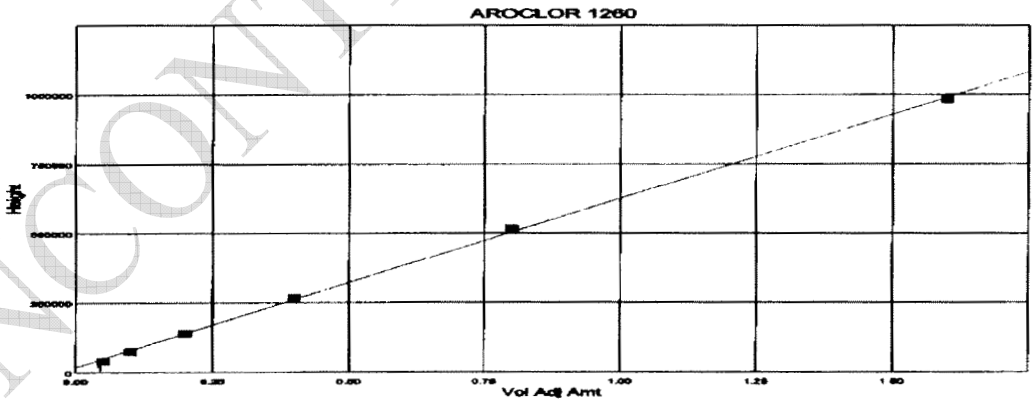


Level Name	Observed X-Value	Calculated X-Value	Delta	%Diff.	Observed Y-Value	Calculated Y-Value	Delta	%Diff.
1	0.050000	0.030468	0.019532	64.108	24989.868	31983.401	-6993.639	-21.866
2	0.100000	0.092697	0.007303	7.879	47271.013	49886.007	-2614.994	-5.242
3	0.200000	0.208577	-0.008577	-2.713	87688.238	85691.218	1997.019	2.330
4	0.400000	0.424569	-0.024569	-5.787	166098.724	157301.641	8797.083	5.592
5	0.800000	0.804923	-0.004923	-0.612	302885.168	300522.497	1762.681	0.587
6	1.600000	1.591766	0.008234	0.517	584015.925	586964.178	-2948.253	-0.502

Fit Analysis Output For Method File: "C:\TC4\GC144\AB183\PCBAA.MTH"
Component Name: "AROCLOR 1260"
Date: 11/2/07 Time: 14:18

Curve Parameters:

Curve #1 : 1st Order
Weighting Factor = 1.0 (No Weighting) r^2 = 0.998451
Calibration Curve = (18219.910667) + (607243.679103)X



Level Name	Observed X-Value	Calculated X-Value	Delta	%Diff.	Observed Y-Value	Calculated Y-Value	Delta	%Diff.
1	0.050000	0.036571	0.013429	36.719	40427.606	48582.095	-8154.488	-16.785
2	0.100000	0.092478	0.007522	8.134	74376.448	78944.279	-4567.831	-5.786
3	0.200000	0.200880	-0.000880	-0.488	140263.845	139668.646	595.199	0.426
4	0.400000	0.411739	-0.011739	-2.851	268245.919	261117.382	7128.537	2.730
5	0.800000	0.820798	-0.020798	-2.534	516644.454	504014.854	12629.600	2.506
6	1.600000	1.587433	0.012567	0.792	982178.780	989809.797	-7631.017	-0.771

UNCONTROLLED COPY

Approved By: [signature] 8-21-09 QA Officer Approved By: [signature] 8/21/09 Area Supervisor

gr03128 2.5.doc

Attachment 21.4
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0705430 Page 1 of 1

Printed: 9/14/2007 4:20:19PM

Semivolatiles GC, Water, 3510C Liquid-Liquid Extraction

Surrogate #1 = 7040593 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0705288	8082 PCBs (std 7 aroclors)	0705322	8082 PCBs (std 7 aroclors)		

Lab Number	*Contain*	*Prepared*	*By*	*Initial (mL)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0705430-BLK1		May-18-07 09:34	ASC	1000	2	1000				BLANK	
0705430-BS1		May-18-07 09:34	ASC	1000	2	1000		6120832	1000	LCS	
0705288-01	D	May-18-07 09:34	ASC	1000	2	1000				[redacted]	
0705322-01	H	May-18-07 09:34	ASC	1060	2	1000				[redacted]	
0705322-02	I	May-18-07 09:34	ASC	1060	2	1000				[redacted]	
0705322-03	E	May-18-07 09:34	ASC	1060	2	1000				[redacted]	
0705322-04	G	May-18-07 09:34	ASC	1060	2	1000				[redacted]	
0705322-05	E	May-18-07 09:34	ASC	1060	2	1000				[redacted]	

Comments	Analyst Initials

bch_TriMatrix.rpt

Approved By: _____ 8-21-09 **QA Officer**

Approved By: _____ 8/21/09 **Area Supervisor**

Attachment 21.5
Analysis Sequence Report Example

TriMatrix Laboratories, Inc. ANALYSIS SEQUENCE **7052932** Page 1 of 3 Printed: 9/14/2007 4:24:07PM

Semivolatiles GC, Water, May-29-07

Instrument = 144, Calibration = 7E04002

Sequence Analyses:
608 PCBs (std 7 aroclors)
608 PCBs (Tonawanda)
8082 PCBs (master list)
8082 PCBs (std 7 aroclors)

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID*	*Client QC Type*	*Extraction Comments*
7052932-ICV1	QC		7030266		INITIAL CAL CHECK	
7052932-ICV2	QC		7030946		INITIAL CAL CHECK	
7052932-ICV3	QC		7040212		INITIAL CAL CHECK	
7052932-ICV4	QC		7030269		INITIAL CAL CHECK	
7052932-ICV5	QC		7030271		INITIAL CAL CHECK	
0705286-04	8082 PCBs (master list)	A 02			[redacted]	[redacted]
0705392-01	8082 PCBs (std 7 aroclors)	C 02			[redacted]	[redacted]
0705433-BLK2	QC				BLANK	
0705433-BS2	QC				LCS	
0705430-BLK1	QC				BLANK	
0705430-BS1	QC				LCS	
0705288-01	8082 PCBs (std 7 aroclors)	D 01			[redacted]	
0705322-01	8082 PCBs (std 7 aroclors)	H 01			[redacted]	
0705322-02	8082 PCBs (std 7 aroclors)	I 01			[redacted]	
0705322-03	8082 PCBs (std 7 aroclors)	E 01			[redacted]	
7052932-CCV1	QC		7030271		CALIBRATION CHECK	
0705322-04	8082 PCBs (std 7 aroclors)	G 01			[redacted]	
0705322-05	8082 PCBs (std 7 aroclors)	E 01			[redacted]	
7052932-CCV2	QC		7030271		CALIBRATION CHECK	
0705651-BLK1	QC				BLANK	
0705651-BS1	QC				LCS	

Comments: | Analyst Initials:

seq_TriMatrix.rpt

Approved By: [signature] 8-21-09 **QA Officer** **Approved By:** [signature] 8/21/09 **Area Supervisor**

UNCONTROLLED COPY

Attachment 21.6
Sample Page of Instrument Run Logbook

TriMatrix Laboratories, Inc.

Instrument ID: HP5890 SERIES II 144	Date: 4/14/04	Sequence #: AB90	Date Archived, Disk ID
Instrument Settings/Injection Volume	**Breakdown**	**Quantitation Information**	**Routine Maintenance Items**
Column Type: RTXCLP/RTXCLP2	DDT % Breakdown:	Curve Date:	New Column Date:
Injection Volume: 1 uL	Endrin % Breakdown:	Curve Type: Regression or Average CF	Column Clipped: (Yes) No
Program: 1 / 2 / 3 / 4	Total % Breakdown <20%: Pass / Fail	Quantitated By: Curve	New Injection Port Liner: (Yes) No
			New Septa: (Yes) No
			New Syringe: Yes (No)

Analyst	Run ID	File ID	Injection Time	Method	Client	Matrix	Dilution	ICV/CCV Check (85-115)	Sample Notes, Standard Numbers, Analytical Batch Information, etc.
JTAM	INST BLK	98	19:48	PC04144A/B		QC		PASS	
	1232	99	20:06						GC36-1
	1242	100	20:24						GC36-3
	1248	101	20:42						GC36-5
	1221/1254	102	21:00						GC36-7
	L1	103	21:18						GC36-10
	L2	104	21:36						
	L3	105	21:54						
	L4	106	22:12						
	L5	107	22:30						
	L6	108	22:48						
	L7	109	23:06						
	LCS	110	23:24						GC37-14
	MPB102L2409A	111	23:42			Soil			
	LFB102	112	0:00						
↓	LFB102 ↓	113	0:18	↓		↓			

Approved By: PD 8-21-09
QA Officer

Approved By: 8/24/09
Area Supervisor

UNCONTROLLED COPY

Attachment 21.7
Method Detection Limit Study Example

TriMatrix Laboratories, Inc.

SEMI-VOLATILE LABORATORY
2007 SOIL
METHOD DETECTION LIMIT VERIFICATION STUDY

Parameter / Compound	Instrument Number	Reference Citation	MDL Result	Units	Date	Analyst	Verification Concentration	MDL/MDL Verification Concentration Difference	MPB Result	MDL Verification Result	MPB/MDL Verification Response Increase	Acceptable (≥3x MPB Response)?
Aroclor 1016	144B	8082	0.0042	mg/kg	4/12/07	JLW	0.01666	3.97	<.01666	0.01301	>3	Yes
Aroclor 1221	144B	8082	0.0060	mg/kg	4/25/07	JLW	0.01666	2.78	<.01666	0.02534	>3	Yes
Aroclor 1232	144B	8082	0.0084	mg/kg	4/12/07	JLW	0.01666	1.98	<.01666	0.02401	>3	Yes
Aroclor 1242	144B	8082	0.0163	mg/kg	4/12/07	JLW	0.01666	1.02	<.01666	0.02405	>3	Yes
Aroclor 1248	144B	8082	0.0038	mg/kg	4/12/07	JLW	0.01666	4.38	<.01666	0.02169	>3	Yes
Aroclor 1254	141B	8082	0.0040	mg/kg	4/12/07	JLW	0.01666	4.17	<.01666	0.02225	>3	Yes
Aroclor 1260	144B	8082	0.0054	mg/kg	4/12/07	JLW	0.01666	3.09	<.01666	0.00921	>3	Yes
Aroclor 1262	144B	8082	0.0052	mg/kg	4/12/07	JLW	0.01333	2.56	<.01333	0.01908	>3	Yes
Aroclor 1268	144B	8082	0.0044	mg/kg	4/12/07	JLW	0.01333	3.03	<.01333	0.02278	>3	Yes

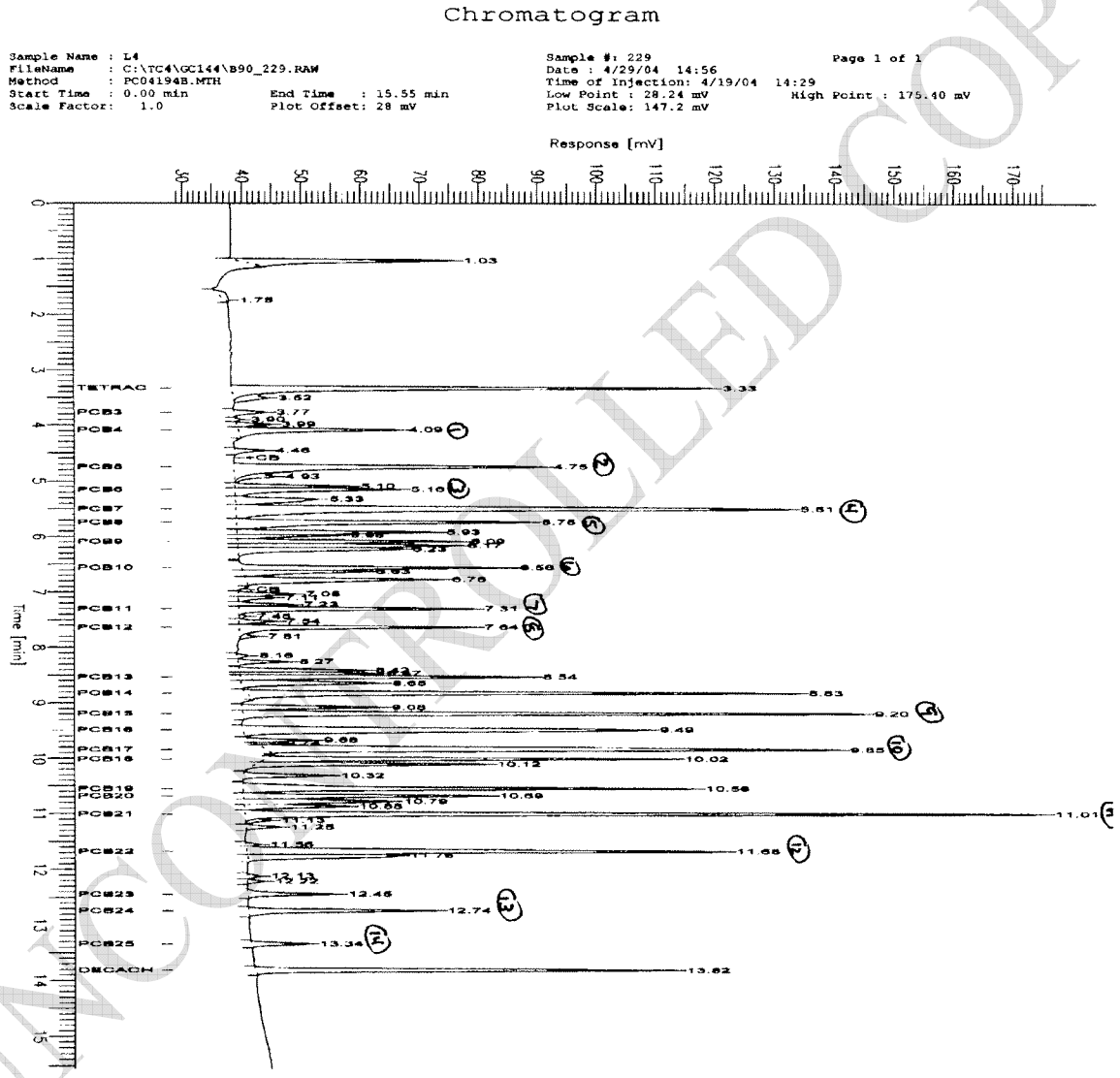
file 8082 SOIL PCB_MDL2007.XLS

revision 2006.14

Approved By: 8-21-09
QA Officer

Approved By: 8/21/09
Area Supervisor

Attachment 21.8
Key Peaks for Aroclor Identification

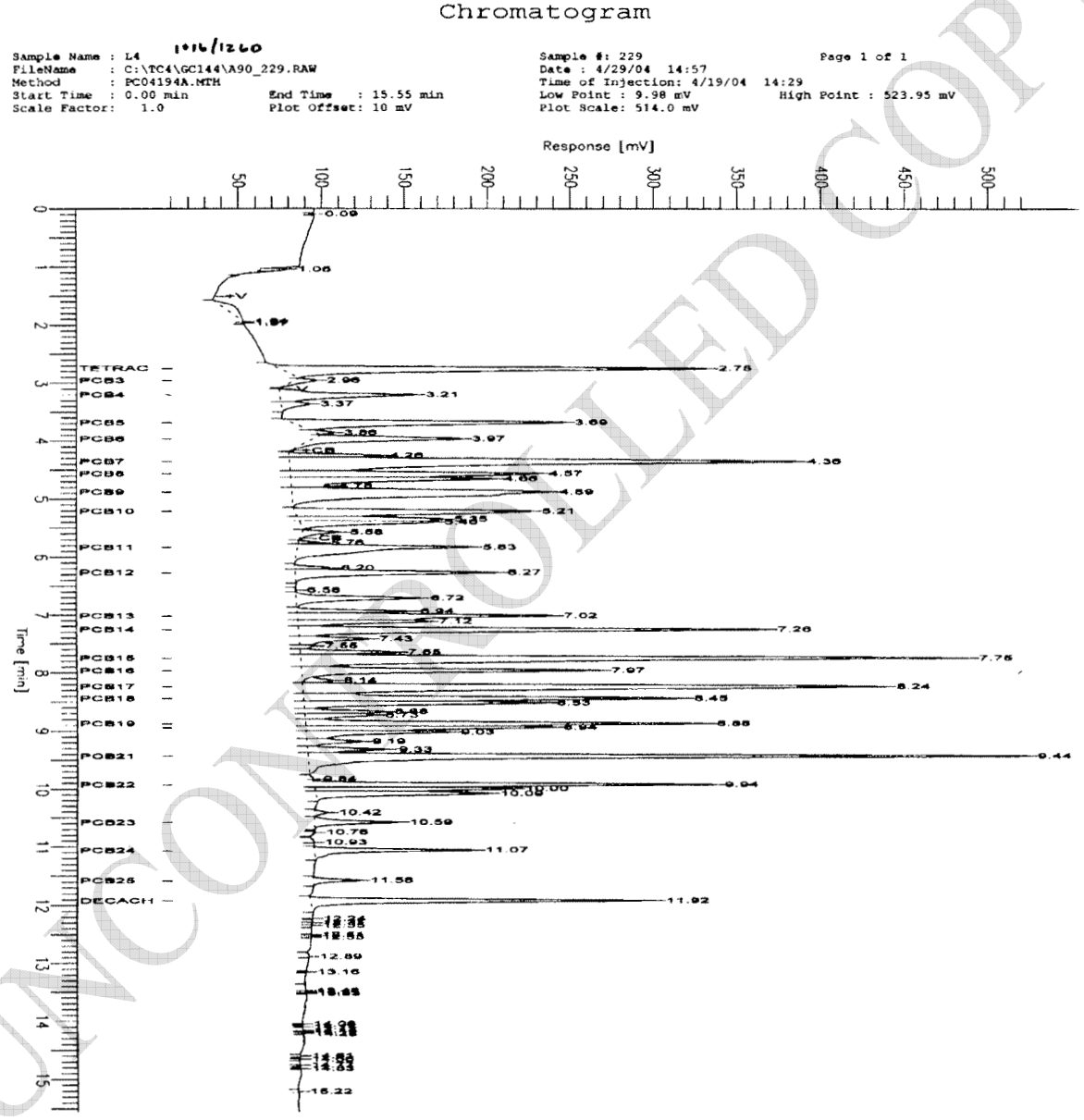


UNCONTROLLED COPY

Approved By: _____ **QA Officer** 8-21-09

Approved By: _____ **Area Supervisor** 8/21/09

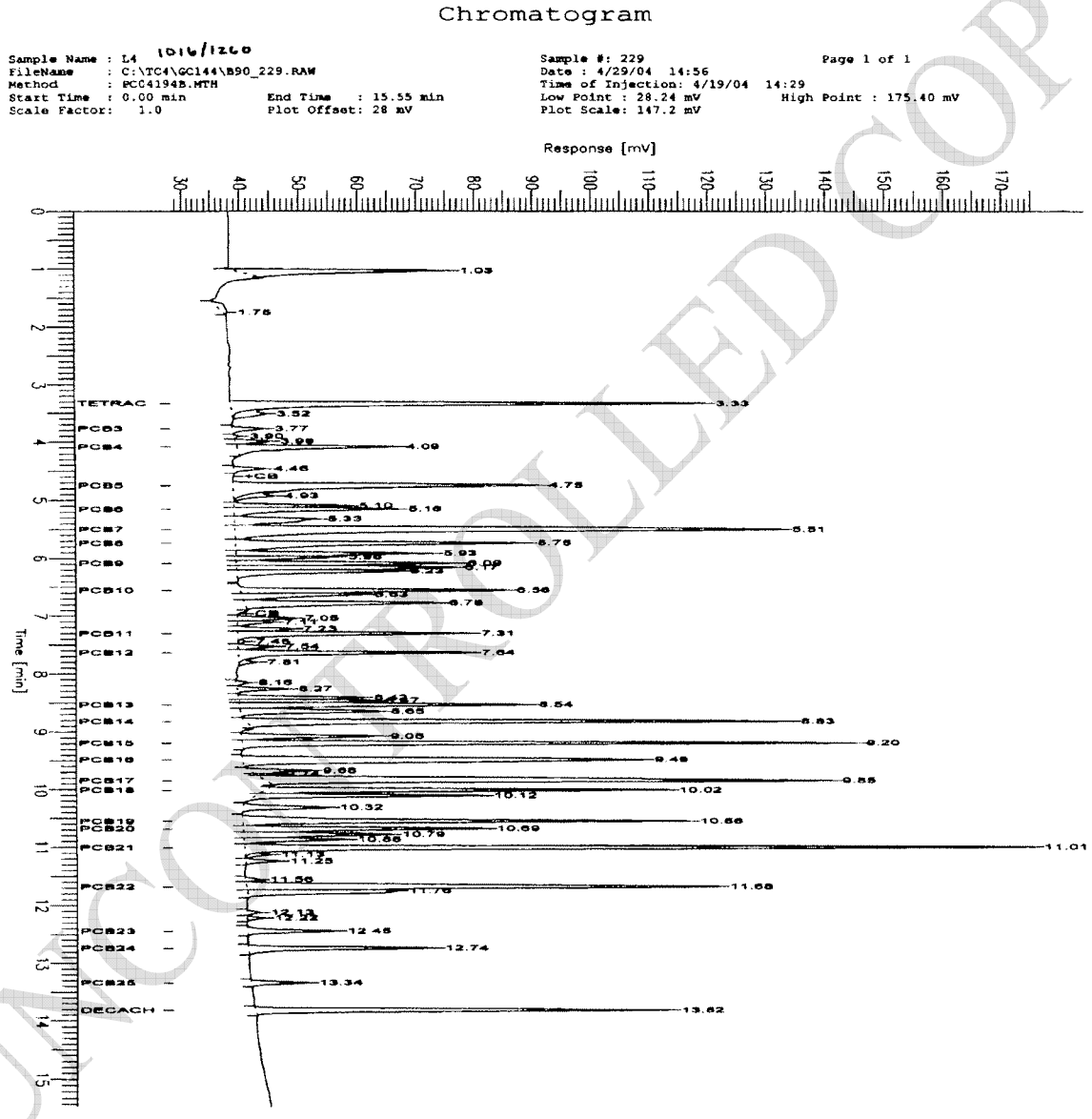
Attachment 21.9
1016/1260 on RTX CLP Column



UNCONTROLLED COPY

Approved By: _____ 8/21/09 **QA Officer** **Approved By:** _____ 8/21/09 **Area Supervisor**

Attachment 21.10
1016/1260 on RTX CLP2 MS Column

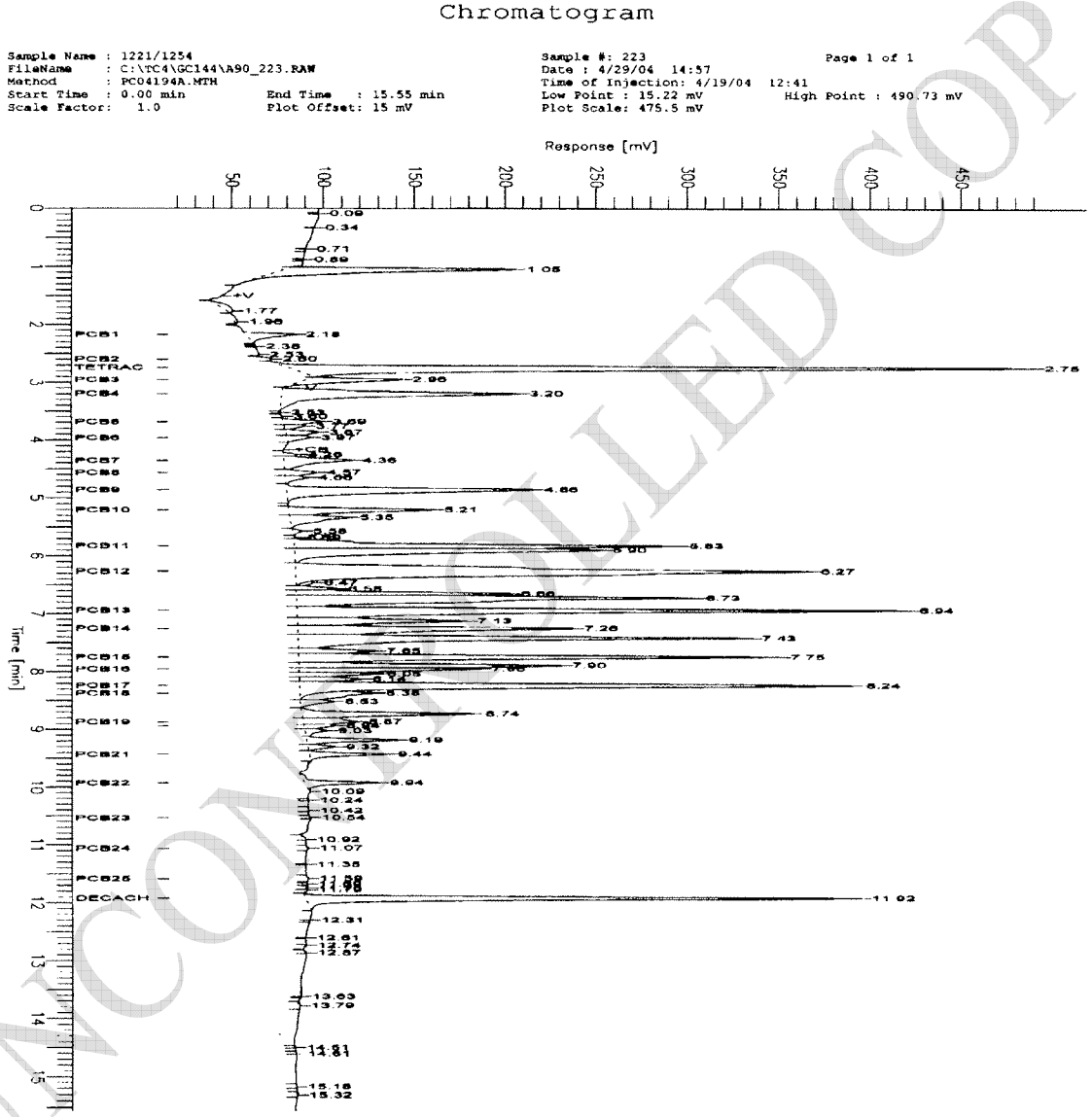


UNCONTROLLED COPY

Approved By: _____ **QA Officer** 8-21-09

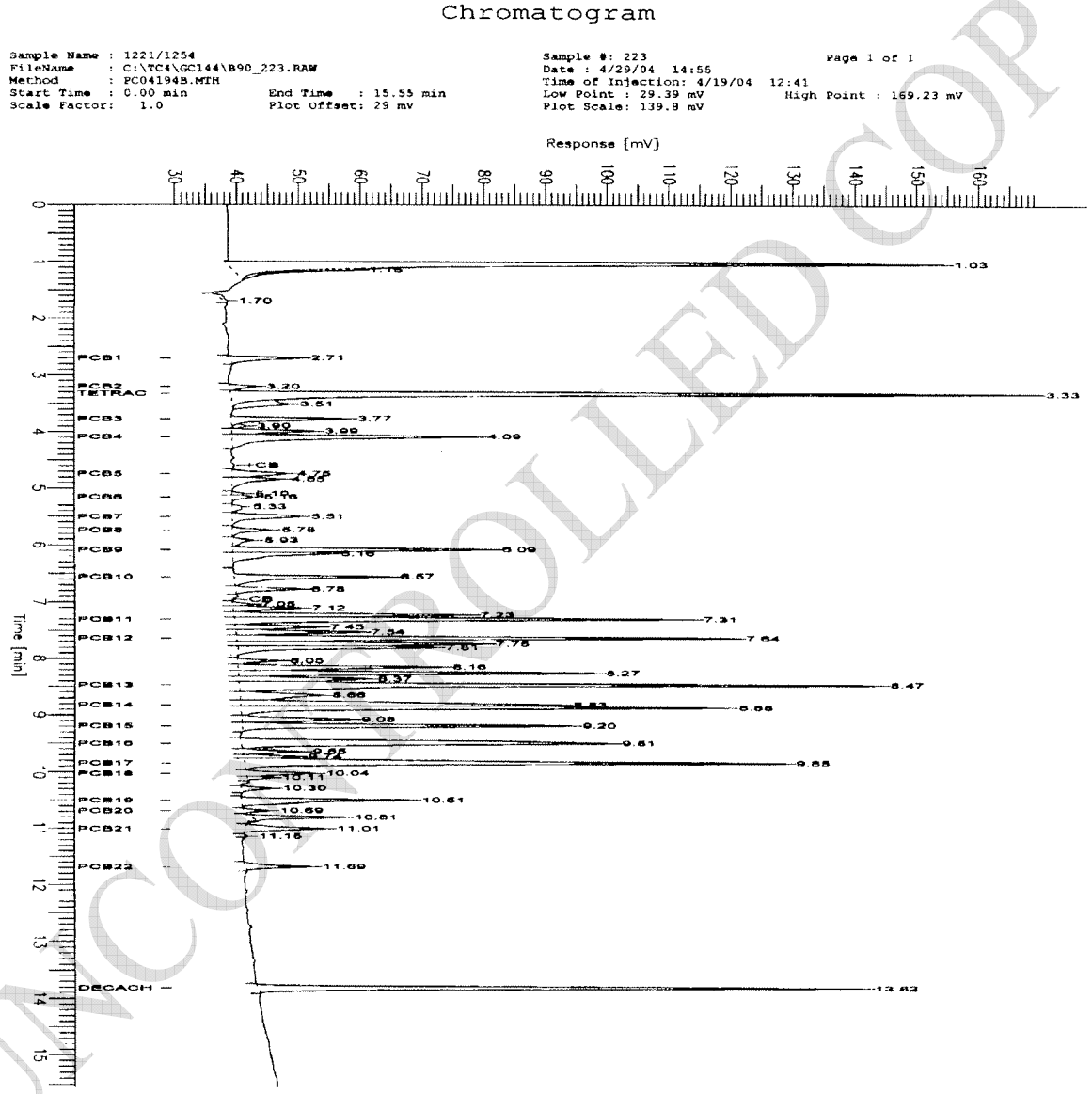
Approved By: _____ **Area Supervisor** 9/2/09

Attachment 21.11
1221/1254 on RTX CLP Column



UNCONTROLLED COPY

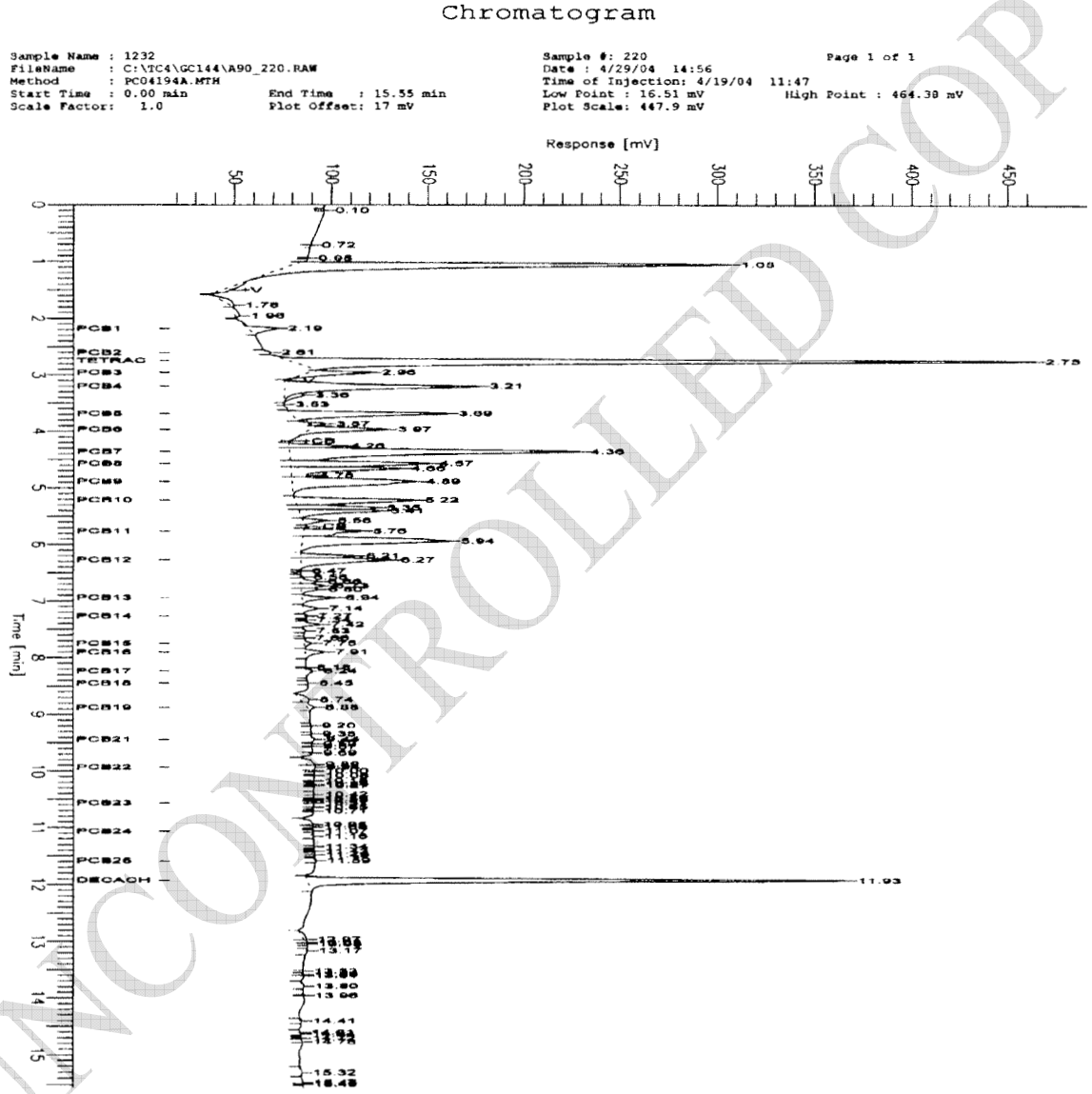
Attachment 21.12
1221/1254 on RTX CLP2 Column



UNCONTROLLED COPY

Approved By: _____ 8-21-09 **QA Officer** **Approved By:** _____ 8/21/09 **Area Supervisor**

Attachment 21.13
1232 on RTX CLP Column



UNCONTROLLED COPY

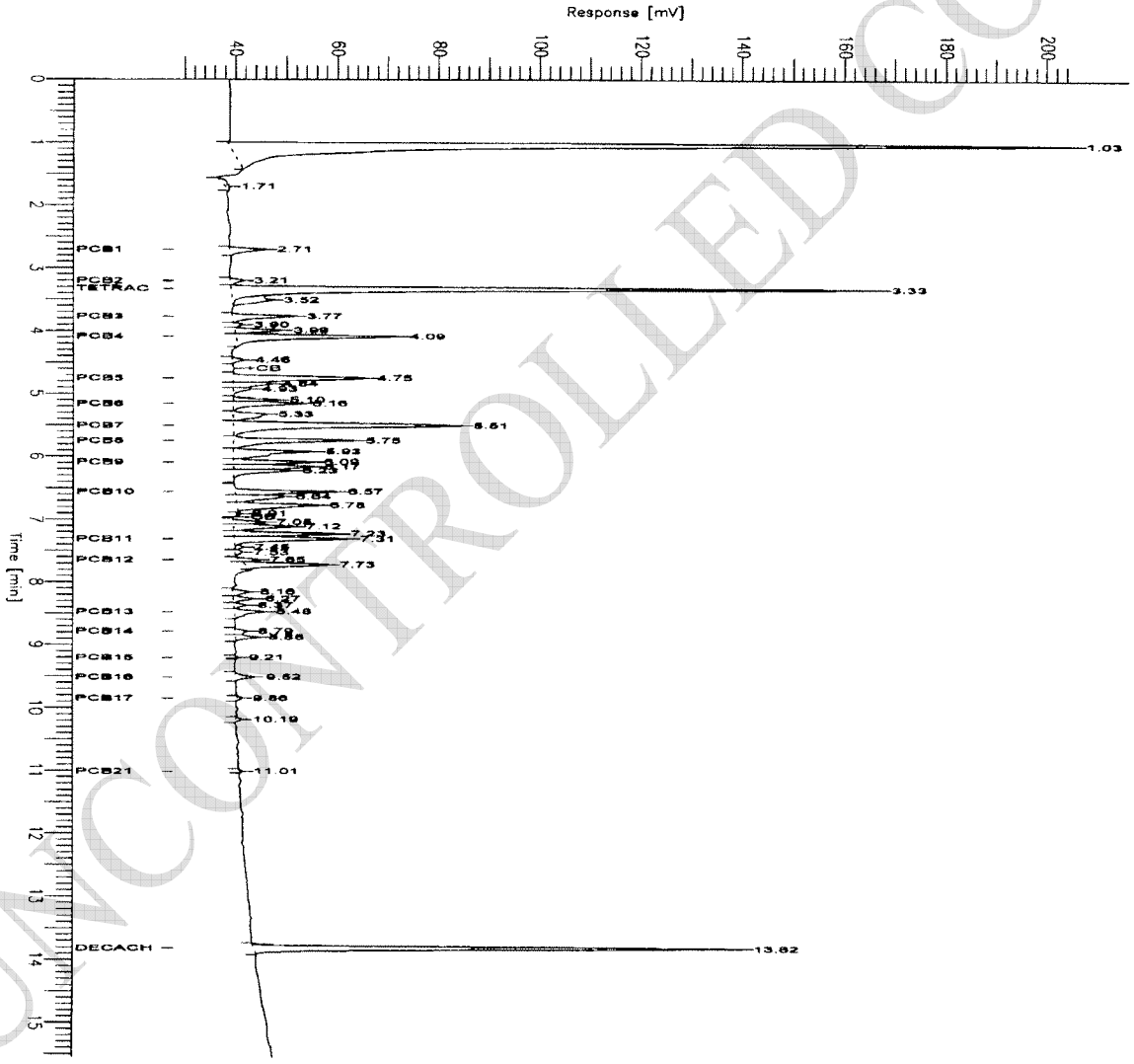
Approved By: _____ 8-21-09 **QA Officer** **Approved By:** _____ 8/21/09 **Area Supervisor**

Attachment 21.14
1232 on RTX CLP2 Column

Chromatogram

Sample Name : 1232
FileName : C:\TC4\GC144\B90_220.RAW
Method : PC04194B.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 28 mV

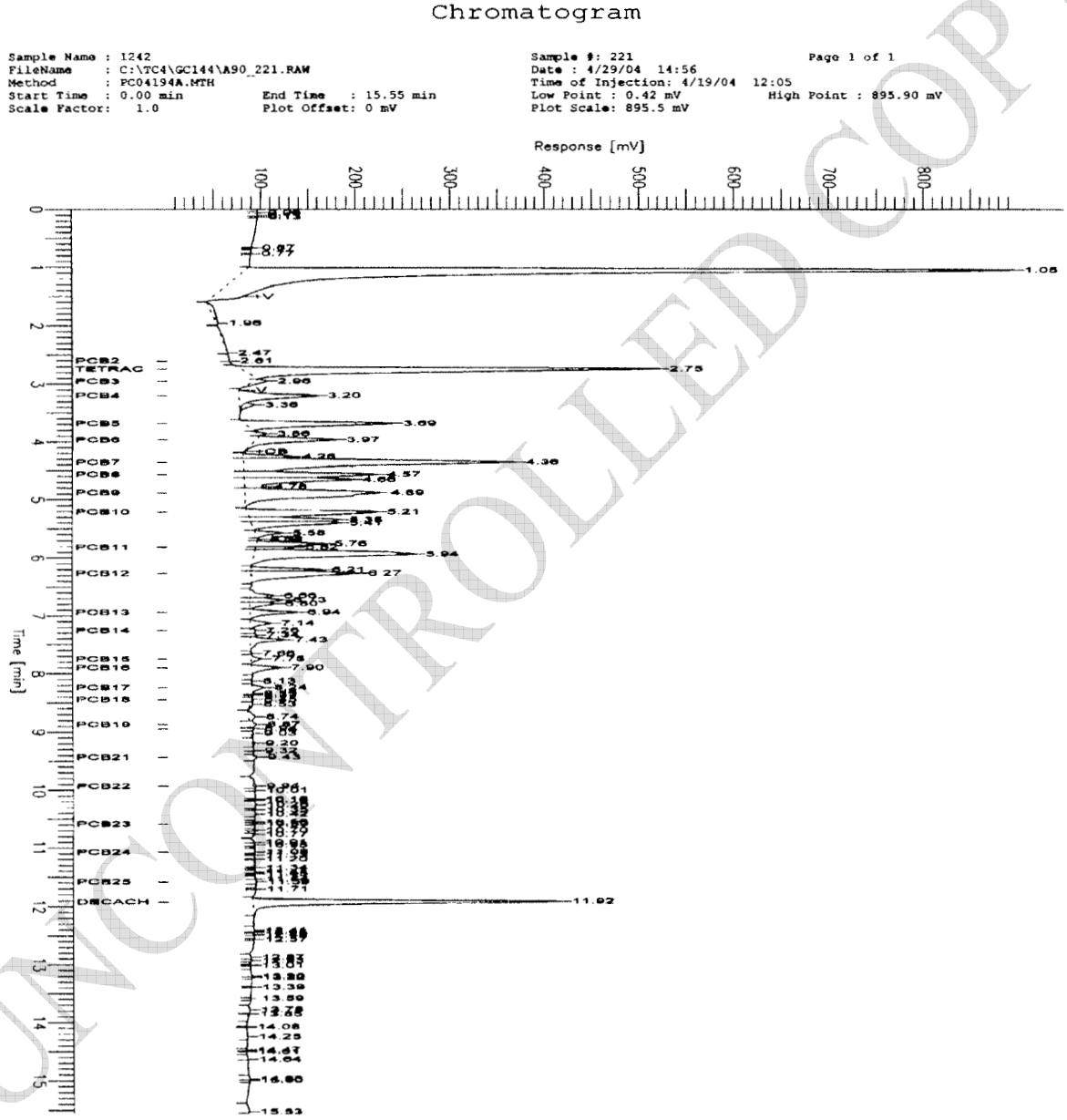
Sample #: 220 Page 1 of 1
Date : 4/29/04 14:55
Time of Injection: 4/19/04 11:47
Low Point : 28.04 mV High Point : 205.58 mV
Plot Scale: 177.5 mV



Approved By: _____ 8-21-09 **Approved By:** _____ 8/21/09
QA Officer **Area Supervisor**

UNCONTROLLED COPY

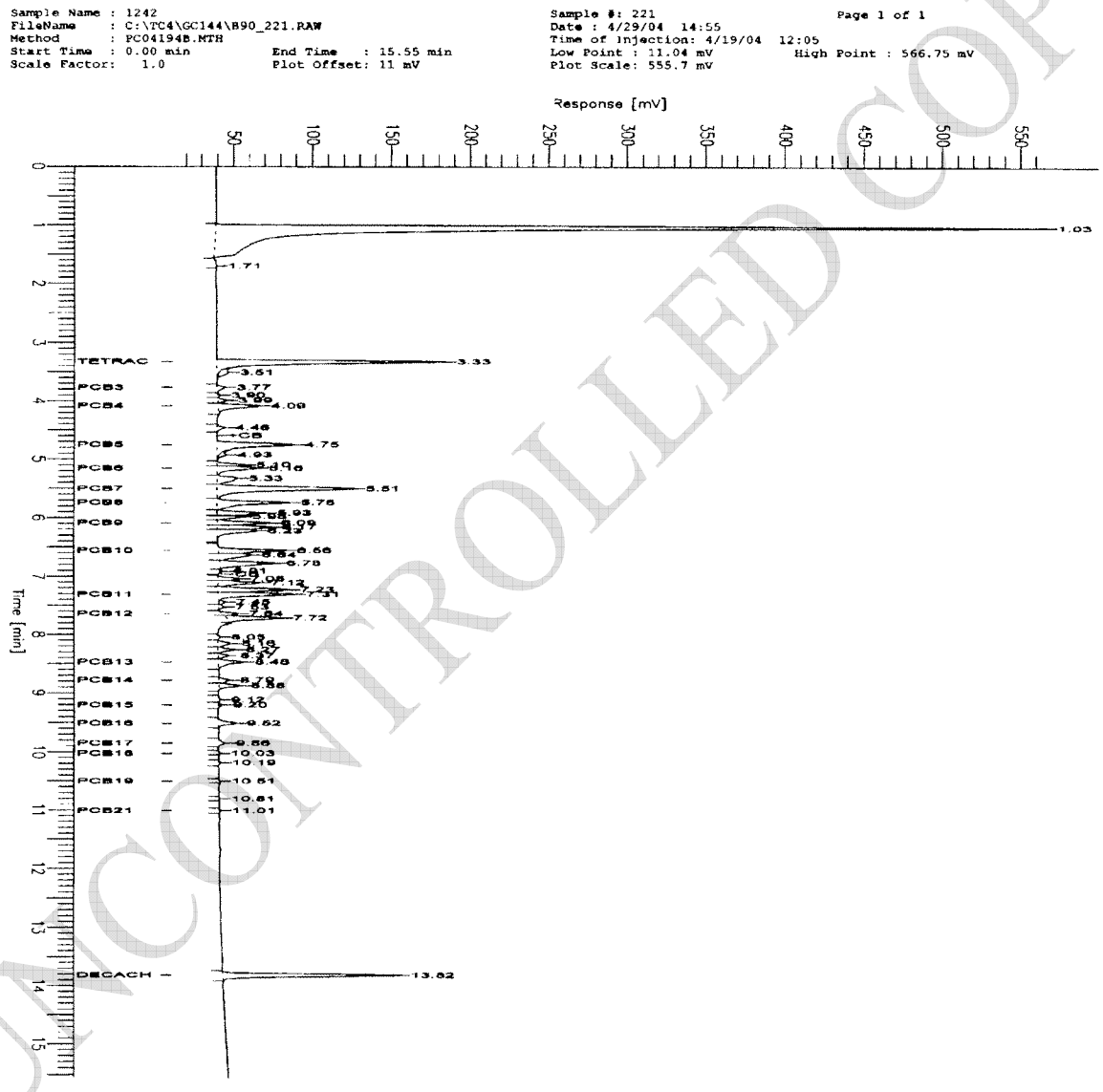
Attachment 21.15
1242 on RTX CLP Column



UNCONTROLLED COPY

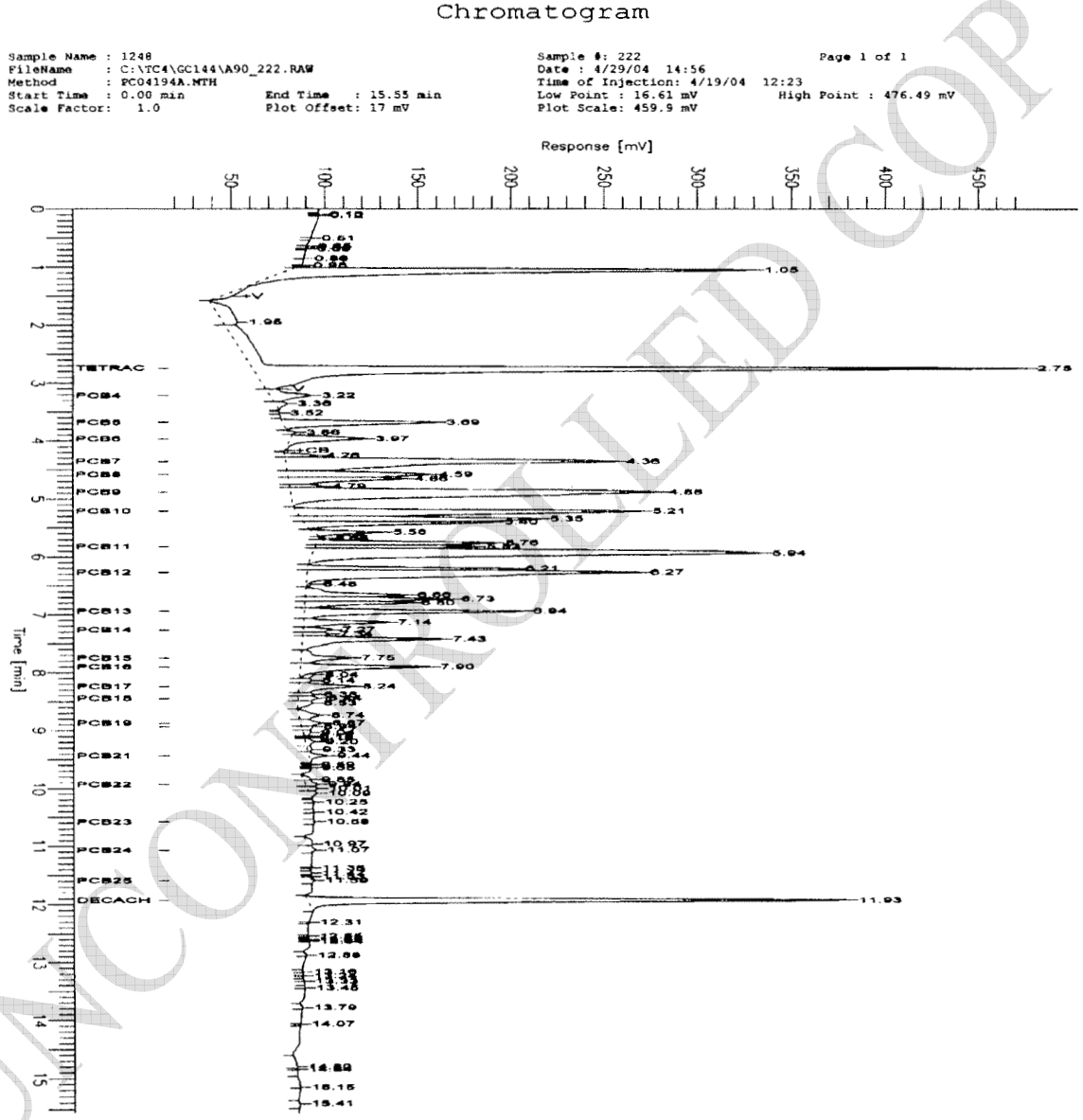
Attachment 21.16
1242 on RTX CLP2 Column

Chromatogram



UNCONTROLLED COPY

Attachment 21.17
1248 on RTX CLP Column

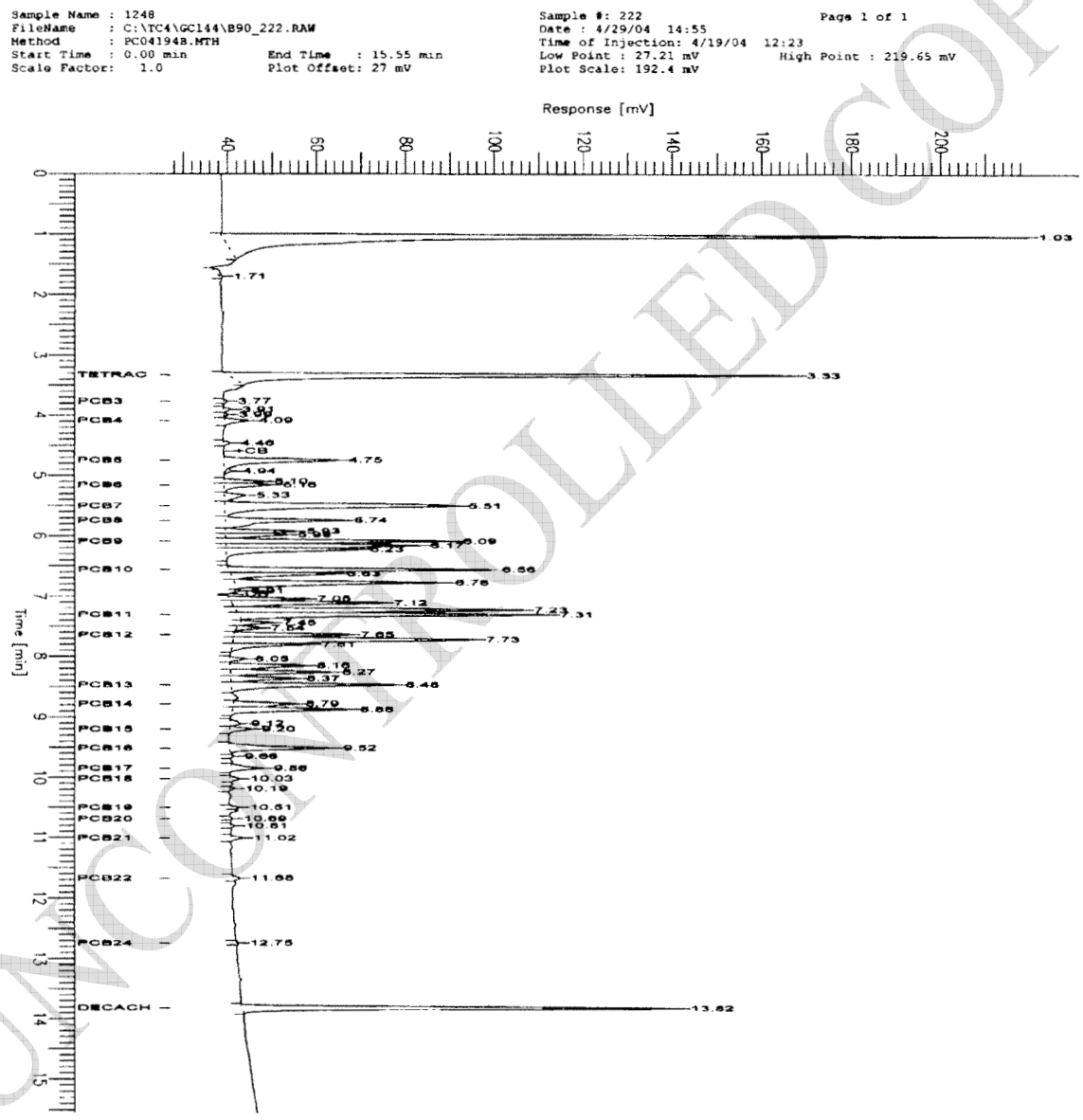


UNCONTROLLED COPY

Approved By: 10 8-21-09 **QA Officer** **Approved By:** 8/21/09 **Area Supervisor**

Attachment 21.18
1248 on RTX CLP2 Column

Chromatogram

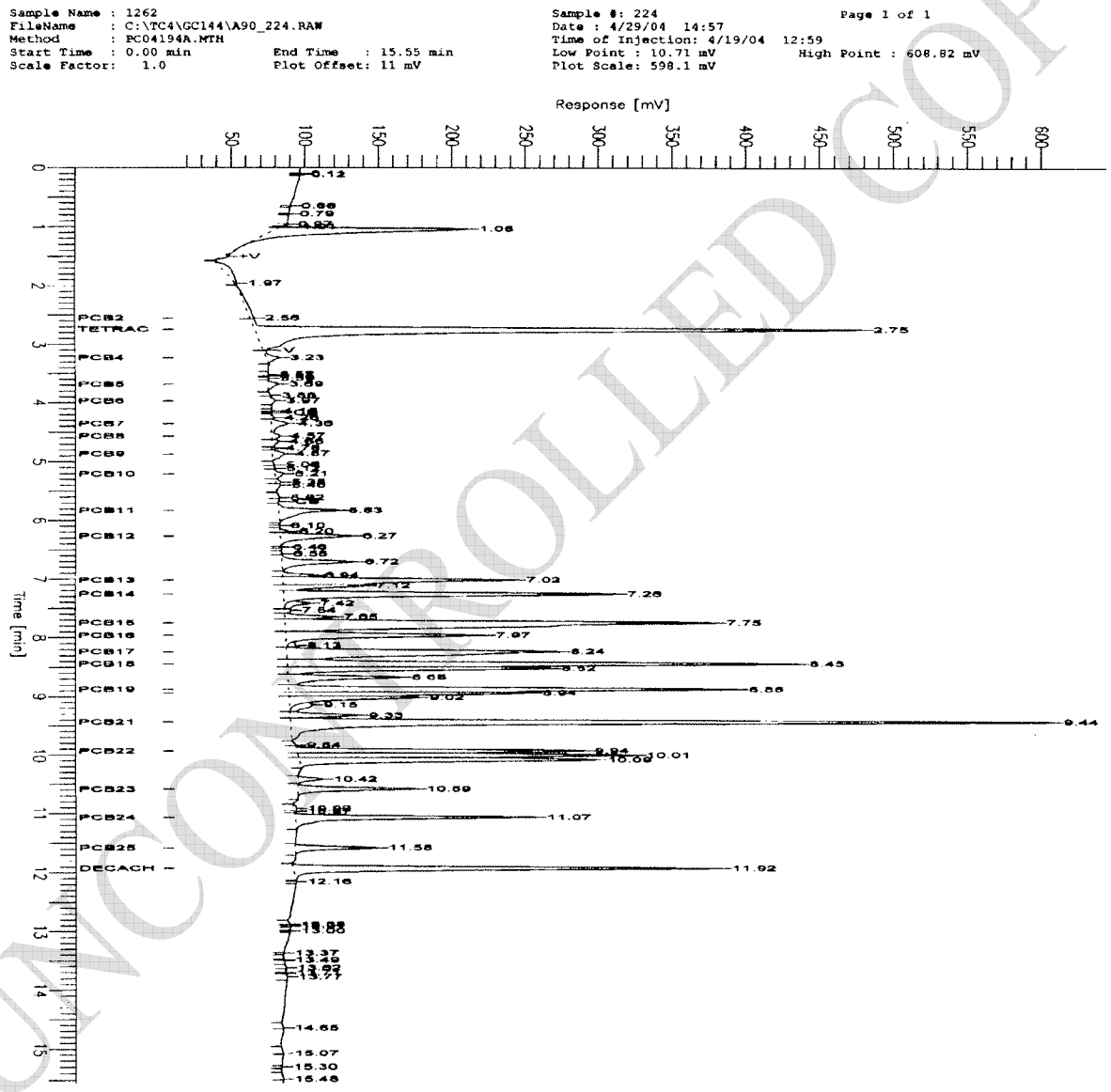


Approved By: _____ 8/21/09 **Approved By:** _____ 8/21/09
QA Officer **Area Supervisor**

UNCONTROLLED COPY

Attachment 21.19
1262 on RTX CLP Column

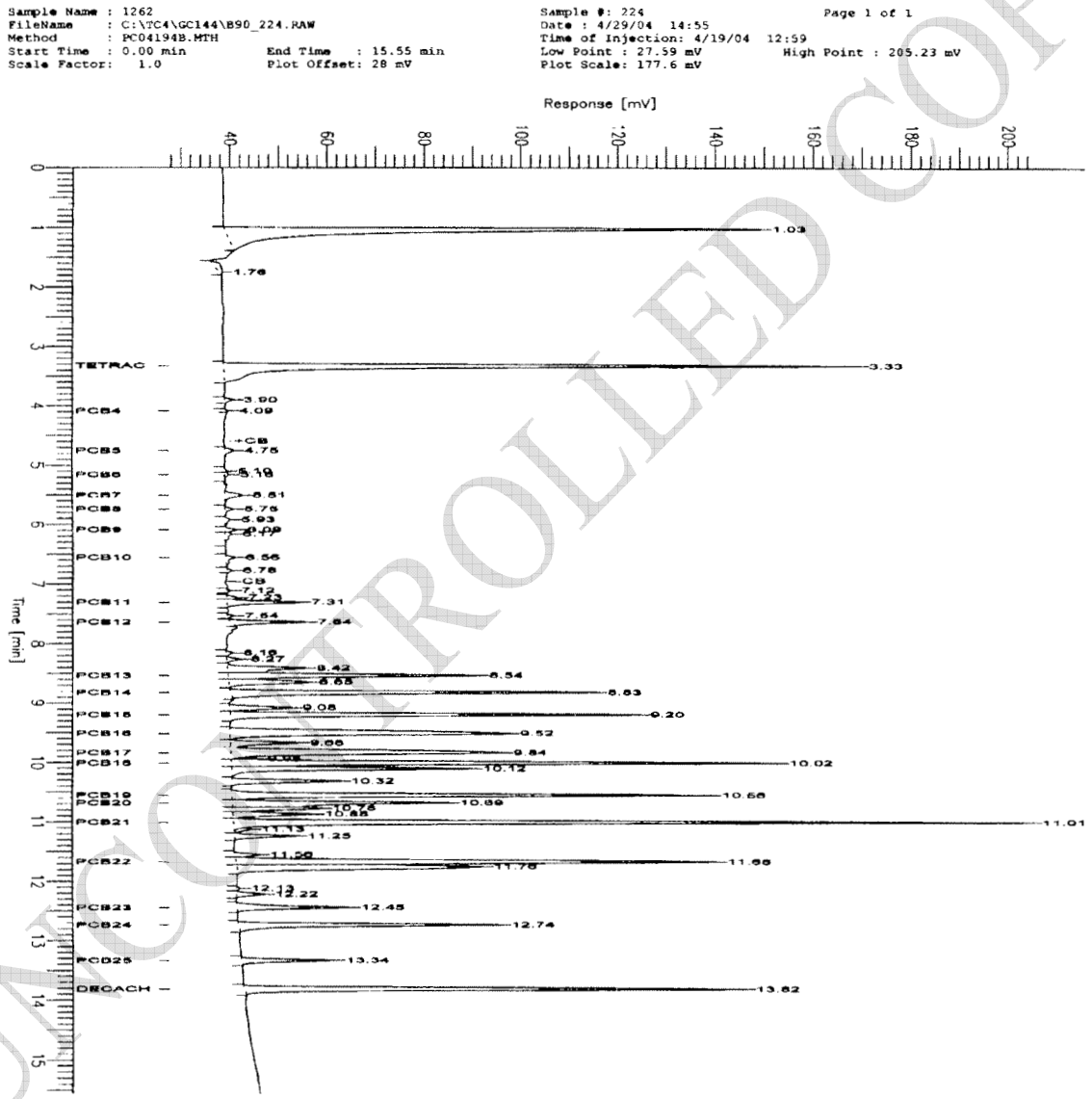
Chromatogram



UNCONTROLLED COPY

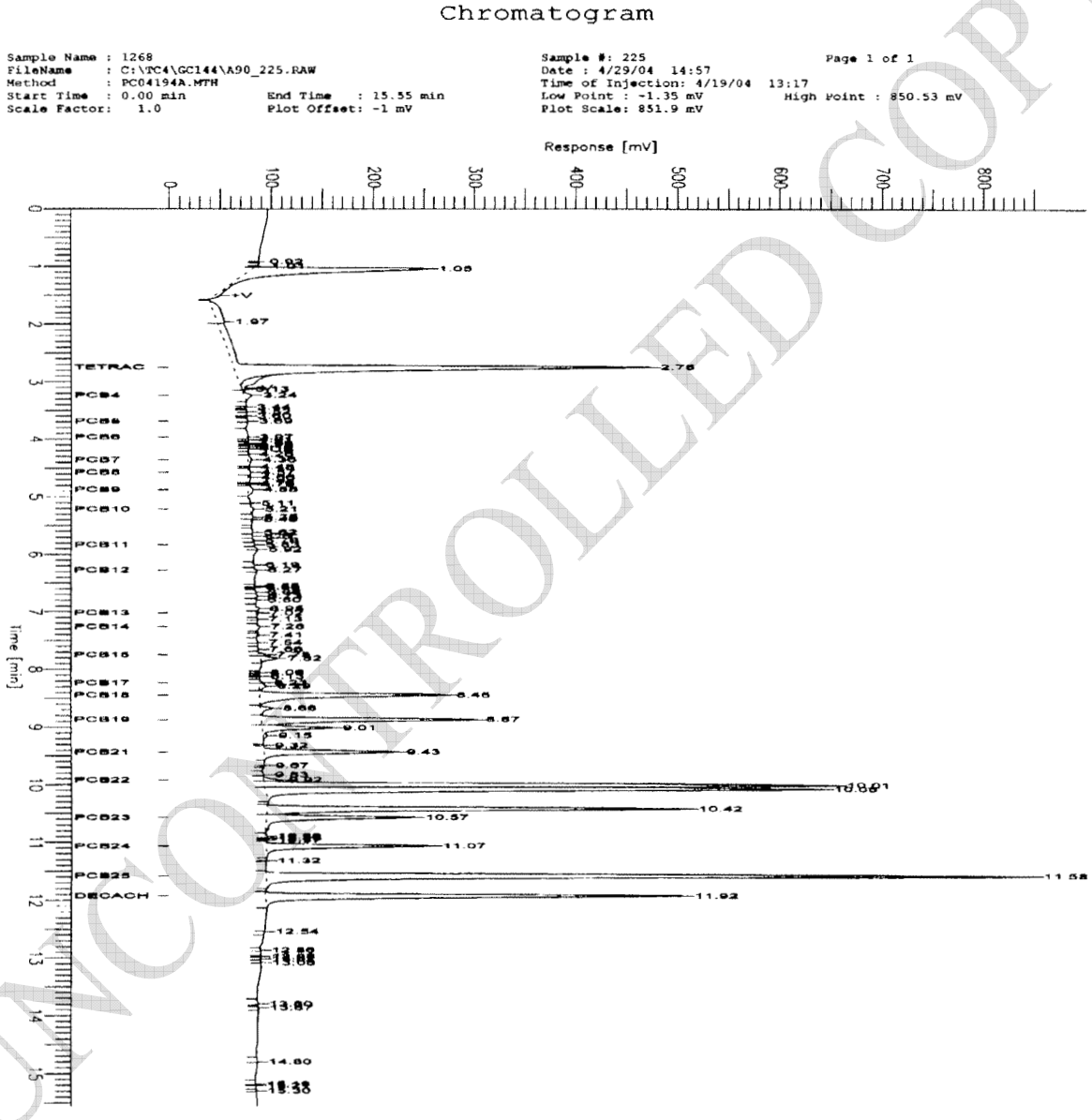
Attachment 21.20
1262 on RTX CLP2 Column

Chromatogram



UNCONTROLLED COPY

Attachment 21.21
1268 on RTX CLP Column

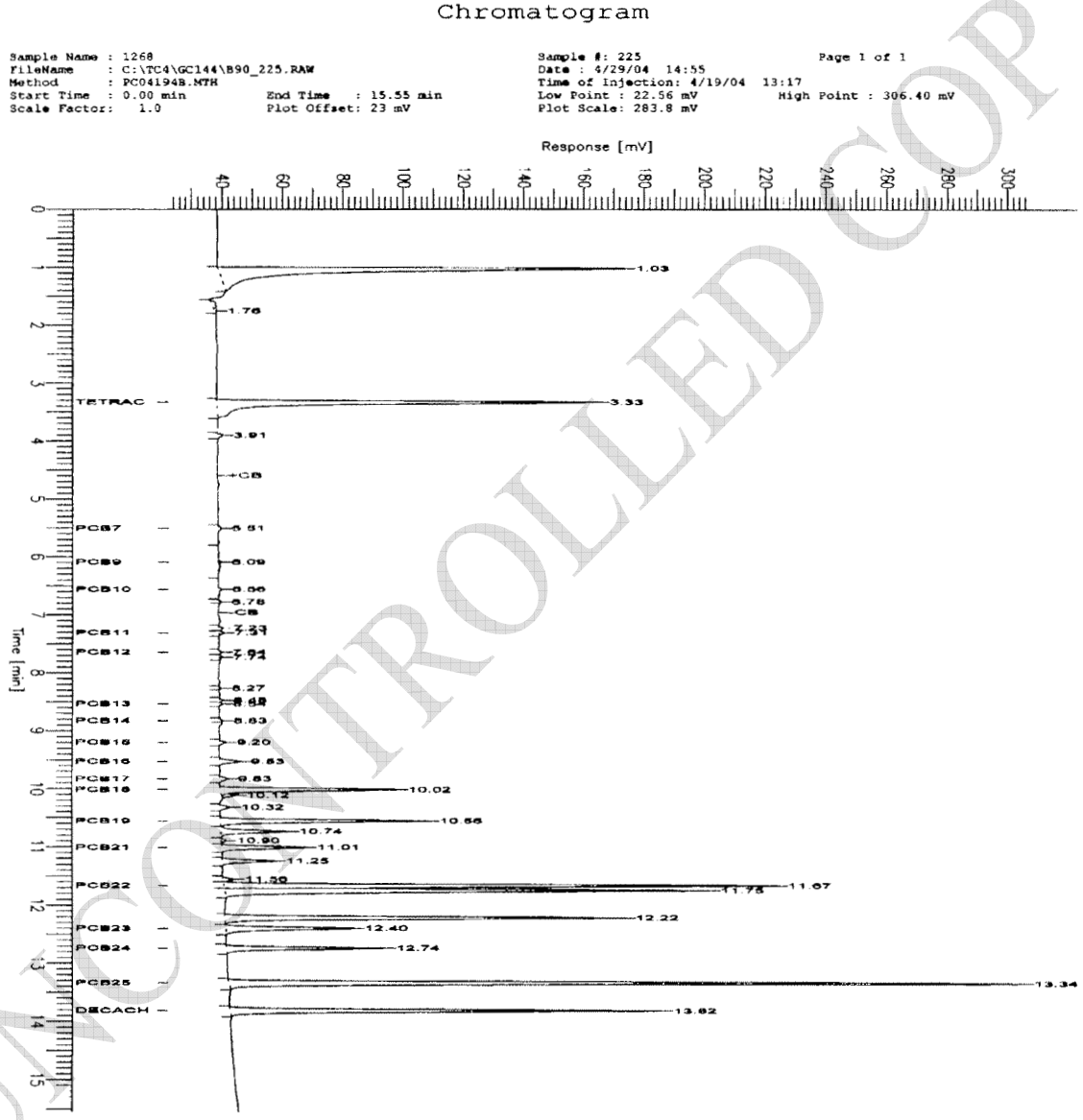


UNCONTROLLED COPY

Approved By: _____ 8-21-09 **QA Officer**

Approved By: _____ 8/21/09 **Area Supervisor**

Attachment 21.22
1268 on RTX CLP2 Column



UNCONTROLLED COPY

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry

EPA Method 625
SW-846 Method 8270C

APPROVALS:

Operations Manager: Jeff P. Glaser Date: 1/23/09

QA Officer: Tom C. Boocher Date: 1-22-09

Area Supervisor: Janet M. Kudirka Date: 1/23/09

Procedure Number: GR-04-103
Revision Number: 5.5

Date Initiated: 12/9/98
Effective Date: 2/20/09

Date Revised: 1/22/09
Pages Revised: All

By: Janet M. Kudirka
Total Number of Pages: 46

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is used to determine the concentration of semi-volatile organic compounds in extracts of various solid waste matrices, soils, groundwaters, and wastewaters.

1.2 This procedure is used to quantify most basic, neutral, and acid extractable organics that are soluble in methylene chloride, and capable of elution without derivatization as sharp peaks from a gas chromatograph, fused-silica capillary column. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols. Refer to Table 1 for routine compounds, retention times, primary and secondary quantitation ions, internal standards, and surrogates. Table 1A lists additional analytes that are amenable to this procedure.

1.3 The following compounds may require special treatment. Benzidine can be subject to oxidative loss during solvent concentration. The chromatography must be monitored for peak broadening and tailing. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction with acetone, and photochemical decomposition. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. 1,2-diphenylhydrazine also decomposes in the injection port into azobenzene. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling materials.

1.4 Default reporting limits for clean extracted samples using 1000 mL or 30 g concentrated to 10 mL and analyzed by this procedure are listed in Tables 2 and 2A. Reporting limits will be proportionately higher depending upon the sample matrix, the preparation technique and extract dilutions. If a client requires lower reporting limits than those in Tables 2 and 2A, the final extract volume may be concentrated to 1.0 mL

1.5 This procedure is restricted to use by or under the supervision of analysts experienced in gas chromatography/mass spectrometry, and skilled in mass spectra interpretation. Each analyst must demonstrate the ability to generate acceptable results.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 3, December 1996

2.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids

2.3 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. B, Definition and Procedure for the Determination of the Method Detection Limit

3.0 SUMMARY OF PROCEDURE

3.1 Before analysis, samples must be extracted using appropriate sample preparation and cleanup techniques. Waters will be extracted using TriMatrix SOP GR-09-101. Soils will be extracted using TriMatrix SOP GR-09-103. Extracts are analyzed by injection into a gas chromatograph, with mass spectrometric identification and detection of compounds. Quantitation is made by comparison with standards.

3.2 Wastewater sample analysis for NPDES permits under the Clean Water Act follows USEPA Method 625, requiring the following modifications to this procedure.

3.2.1 DFTPP tunes and continuing calibration standards are analyzed once every 24 hours instead of every 12.

3.2.2 A 3-point calibration may be used in place of a 6-point calibration. Standard practice, however will generally be to quantify using the 6-point calibration. System Performance Check Compounds (SPCCs) and Calibration Check Compounds (CCCs) are not used. Instead the response factors (RF) for every compound of interest must have <35% RSD to use the average RF for quantitation. If these criteria are not met, a new calibration curve must be constructed.

3.2.3 The continuing calibration standard does not use SPCCs and CCCs. The RF for compounds of interest in the continuing calibration standard that is on the 625 list must be ≤20% difference when compared against the calibration. If not, a new continuing calibration standard will be run with acceptable percent differences, or a new 3-point or 6-point calibration will be run.

3.2.4 There are no criteria for internal standard areas or retention times in Method 625. Method 8270C criteria will be followed.

3.2.5 Every compound of interest must be in matrix spikes and Laboratory Fortified Blanks, not just the limited list specified in Method 8270C. Laboratory generated windows for surrogate and spike recoveries are calculated as specified in this procedure.

4.0 PARAMETER OR COMPOUND LIST

4.1 Analytes for analysis by Method 8270C or Method 625 are listed in Table 1 and 1A. Other analytes may be determined providing a demonstration of capability study is successfully completed. In addition, QC criteria described for Table 1 and 1A compounds must be considered the minimum acceptable for additional analytes.

5.0 REFERENCED SOPs

5.1 TriMatrix Laboratories SOP GR-09-101, *The Extraction of BNAs in Water*, latest revision

5.2 TriMatrix Laboratories SOP GR-09-103, *Extraction of Semi-Volatile BNAs in Soil, Sediment and Sludge*, latest revision

5.3 TriMatrix Laboratories SOP GR-04-101, *Semivolatile Laboratory Mass Spectrometry Corrective Actions*, latest revision

5.4 TriMatrix Laboratories SOP GR-09-104, *Waste Dilution of BNAs,* latest revision

5.5 TriMatrix Laboratories SOP GR-15-102, *Waste Disposal,* latest revision

5.6 TriMatrix Laboratories SOP GR-10-115, *Manual Integrations*, latest revision

5.7 TriMatrix Laboratories SOP GR-10-123, *ELEMENT Data Transfer and Revies,* latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interferences can be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware, leading to discrete artifacts and/or elevated baselines in the total ion current profiles. Do not use plastic materials, including polypropylene squirt bottles. Phthalate esters leaching from plastics can result in false positives. All materials used in extraction and analysis must routinely be demonstrated to be interference-free by running method performance blanks. Rinse all washed glassware with pesticide grade or better methylene chloride immediately before use. Use only high purity reagents (reagent grate or better) and solvents (pesticide grade or better). Solvents used for extraction and analysis must be tested prior to use. Once they are proven to be contaminant-free, large lots are sequestered from the vendor to ensure purity.

6.2 Carryover can occur whenever highly concentrated samples precede the analysis of low or non-detect samples. To reduce carryover, autosampler syringes are rinsed between extract injections. Whenever possible, the analyst should analyze a reagent blank immediately after a high level sample. If carryover is suspected, all affected samples must be re-analyzed.

6.3 Corrective actions for interference and contamination are outlined in TriMatrix SOP GR-04-101, *Semivolatile Laboratory Mass Spectrometry Corrective Actions*, latest revision.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the Laboratory Safety Manual and Chemical Hygiene Plan. Safety glasses are mandatory in all laboratory areas, except when entering data at computer terminals.

7.2 Methylene chloride and most target analytes, surrogates, and spikes are highly toxic and suspected carcinogens. Every precaution must be taken to avoid dermal contact, ingestion and inhalation. Wear appropriate protective clothing and disposable gloves when handling any of the above compounds. Prepare standards and extracts for analysis under a fume hood.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 To achieve the reporting limits in Tables 2 and 2A, a minimum 30 g of soil or 1000 mL of aqueous sample must be collected and extracted. Additional sample volume is necessary if matrix spikes are required.

8.2 Samples should be collected in amber glass jars, with PTFE-lined screw-cap lids. These jars must meet or exceed current USEPA cleanliness standards. Plastic containers and/or plastic-lined lids must NOT be used due to potential phthalate ester contamination.

8.3 Samples must be stored away from light, at 4° C ± 2° C. Store extracts away from light and ≤ -10° C.

8.4 Analysis must be performed within 40 days of sample extraction.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Gas chromatograph/mass spectrometer system – this procedure is applicable to either of the systems described in 9.1.1 or 9.1.2.

9.1.1 Varian Saturn II ion trap mass spectrometer is set up to scan from 40-450 mass units twice per second, using 70 volts (nominal) electron energy in the electron impact mode. The spectrometer produces a mass spectrum that meets all criteria in Table 3, when 5.0 ng of decafluorotriphenylphosphine (DFTPP) is injected onto the analytical column.

9.1.1.1 Ion Trap operating conditions:

- Electron energy: 70 volts (nominal)
- Mass range: 40-450 amu
- Scan time: 820 amu/second = 2 scan/second
- Source temperature: 190° C
- Transfer line temperature: 250° C

9.1.1.2 Varian 3400 GC operating conditions:

- Column Temperature Program: 45° C for 1.5 minutes, then to 200° C at 10°C/minute, then to 315° C at 25° C/minute, hold at 315° C until benzo[g,h,i]perylene has eluted.
- SPI Injector Temperature Program: 45° C for 0.5 minute, then to 300° C at 100°C/minute.
- Sample volume: 1 uL
- Carrier Gas: ultra high purity helium at 6 psi (~ 1 to 2 mL/minute)

9.1.2 The Agilent 5973 Mass Selective Detector (MSD) is set up to scan from 40-550 mass units at least twice per second, using 70 volts (nominal) electron energy in the electron impact mode. The detector produces a mass spectrum that meets all the criteria in Table 3, when 5.0 ng of decafluorotriphenylphosphine (DFTPP) is injected onto the analytical column.

9.1.2.1 MS operating conditions:

- Electron energy: 70 volts (nominal)
- Mass range: 40-550 amu
- Scan time: sampling rate of 3 = 2.89 scan/second
- Source temperature: 230° C
- Transfer line temperature: 280° C

9.1.2.2 Agilent 6890 GC operating conditions:

- Column Temperature Program: 45° C for 1.5 minutes, then to 200° C at 10°C/minute, then to 315° C at 25° C/minute, hold at 315° C for 4.0 minutes
- Sample volume: 1 uL
- Carrier Gas: ultra high purity helium at 1.0 mL/minute in constant flow mode

9.1.3 Injector

9.1.3.1 A Septum Programmable Injector (SPI), temperature programmed. The injector is capable of sub-ambient initial injector temperatures, followed by rapid heating up to 315° C, and is used for Ion Trap applications.

9.1.3.2 A Split/splitless injector in the splitless mode held at 250° C, is used for the Mass Selective Detector (MSD).

9.1.4 Fused Silica Capillary Columns

9.1.4.1 A 30 m x 0.32mm i.d. 1.0 um film thickness silicon coated DB-5MS or equivalent, is used for the Ion Trap.

9.1.4.2 A 30 m x 0.25 mm, i.d. 0.25 um film thickness silicon coated DB-5MS or equivalent, is used for the Mass Selective Detector (MSD).

9.1.4.3 A 20 m x 0.15 mm. i.d. 0.15 um film thickness silicon coated factorFour VF-5MS is also used for the Mass Selective Detector (MSD).

9.1.5 Data system:

9.1.5.1 A multi-tasking personal computer, capable of the continuous acquisition and storage on magnetic tape or compact disk, of all mass spectra obtained throughout a chromatographic program.

9.1.5.2 The software can search acquired GC/MS data files, for specific mass ions, and can plot ion abundances versus time or scan number. This type of plot is an Extracted

Ion Current Profile (EICP). The software integrates abundances in any EICP between specified time or scan number limits. The 1992 (Ion Trap) or 1998 (MSD) version of the NIST mass spectral library is referenced for identification of unknowns.

9.1.6 Autosampler: A Leap A200SE or equivalent, programmed for 1.0 uL injections with multiple rinses before and after injection.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 When chromatography degrades or sensitivity decreases, routine maintenance consists of cleaning/changing the injection port liner, clipping capillary column, changing injection port septum, and cleaning the source.

10.2 The rough pump oils must be replaced at least annually.

10.3 Check the MSD diffusion pump oil every six months and refill, if necessary.

10.4 Check the Ion Trap turbo pump wick annually and re-oil or replace, if necessary.

10.5 If column performance deteriorates, performance may be brought back by repeated injections of Toluene overnight. If this is ineffective, the column will need replaced.

11.0 CHEMICALS AND REAGENTS

11.1 All chemicals and reagents used must be reagent grade or better, and must be determined free of interferences.

11.2 Acetone, hexane, methylene chloride, and toluene must be pesticide grade or better, and must be determined free of interferences.

12.0 STANDARDS PREPARATION

12.1 Stock calibration standard solutions: Stock solutions are prepared from pure standard materials or purchased as certified solutions.

12.1.1 Commercially purchased stock solutions are used to prepare the majority of working standards. The purchased concentration for stock mixes is typically 2000 mg/L. Solutions arrive at the laboratory sealed in various glass ampules, which are combined during preparation.

12.1.1.1 Stock standards are combined with the surrogate currently being used during sample extraction, to create a 100 mg/L calibration standard in methylene chloride.

12.1.1.2 Diluting from the 100 mg/L standard, make up the 8-point initial calibration at concentrations of 25, 20, 15, 10, 5.0, 2.0, 1.0, and 0.5 mg/L. Due to low response

in the lowest calibration point, prepare extra concentrations of 2,4-dinitrophenol, 4-nitrophenol, benzoic acid and benzidine for a final concentration of 1.0 mg/L to 50 mg/L.

12.1.2 Stock standard solutions from neat compounds are prepared by weighing out each analyte to the nearest tenth of a milligram. The material is dissolved in methylene chloride or other suitable solvent and diluted to volume in a volumetric flask. If compound purity is 96% or greater, the weighed mass is used without correction to calculate stock standard concentration.

12.1.3 Transfer stock standard solutions to screw-cap vials with PTFE-lined lids and store below -5° C, protected from light. Stock standard solutions must be monitored for degradation or evaporation.

12.1.4 All stock standard mixes must be replaced annually or sooner if comparison with quality control checks indicates a problem. Working calibration standards must be replaced every six months or sooner if comparison with quality control checks indicates a problem. Continuing calibration standards must be prepared weekly.

12.2 Internal standard solutions: The six internal standards used are 1,4-dichlorobenzene-d_4, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, naphthalene-d_8, and perylene-d_{12}. The stock standard is prepared in a volumetric flask at 1000 ug/mL by dissolving 100 mg of each compound with a small volume of methylene chloride then diluting to 100 mL. Each 1.0 mL extract analyzed must be spiked with 5.0 uL of the internal standard solution. The resulting concentration of internal standards in extracts is 5.0 ug/mL.

12.3 GC/MS tuning standard: The standard is used for tuning the mass spectrometers and contains 5.0 ng/uL of decafluorotriphenylphosphine (DFTPP), 50 ng/uL of pentachlorophenol, 50 ng/uL of benzidine, and 50 ng/uL of 4,4'-DDT, in methylene chloride. Stock standards for the tuning mixture are purchased from a vendor at 1000 mg/L. Appropriate dilutions for the working concentrations are prepared from these standards. The tuning standard also verifies injection port inertness and monitors GC column performance.

12.4 Surrogate standards: Surrogate solution contains o-terphenyl, 2-fluorophenol, phenol-d6, 2,4,6-tribromophenol, nitrobenzene-d5, and 2-fluorobiphenyl. These are made as follows.

12.4.1 Acid and base compound surrogate solutions are made separately from neat materials:

12.4.1.1 Add 0.5 g of each base-neutral surrogate to a 250 mL volumetric flask, half-filled with methanol. Swirl, shake or sonicate to dissolve, then dilute to volume with methanol.

12.4.1.2 Add 1.0 g of each acid surrogate to another 250 mL volumetric flask, half-filled with methanol. Swirl, shake or sonicate to dissolve, then dilute to volume with methanol.

12.4.1.3 Add 100 mL of each solution to a 2 L volumetric half-filled with methanol. Swirl to mix then dilute to volume with methanol. The finished solution must be checked for accuracy by analyzing on the GC/MS. Once approved, the prep lab and the

mass spec lab must coordinate to be sure the same surrogate mixture is used for calibration and spiking.

12.4.1.4 Transfer the entire 2 L solution to 40 mL amber vials with PTFE-lined screw-cap lids and store in the organic prep laboratory refrigerator at 4 ± 2° C.

Surrogate	Mass (g)	Dilution Volume (mL)	Stock Concentration (mg/L)	Secondary Dilution (mL:mL)	Final working Concentration (ug/mL)
Decafluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
o-terphenyl	0.5	250 in methanol	2000	100:2000	100
Nitrobenzene-d_5	0.5	250 in methanol	2000	100:2000	100
2-Fluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
2,4,6-Tribromophenol	1.0	250 in methanol	4000	100:2000	200
Phenol-d_6	1.0	250 in methanol	4000	100:2000	200
2-Fluorophenol	1.0	250 in methanol	4000	100:2000	200

12.4.2 The volume spiked is 100 uL for each 1.0 mL of extract volume, resulting in a concentration of 10 ng/uL of base/neutral surrogates and 20 ng/uL acids.

12.5 Spiking standards applicable to Laboratory Blank Spikes (BS) and Matrix Spikes (MS/MSD): The base/neutral spike solution contains acenaphthene, 1,4-dichlorobenzene, 2,4,-dinitrotoluene, naphthalene, N-nitrosodi-n-propylamine, pyrene, and 1,2,4-trichlorobenzene. The acid spike contains 4-chloro-3-methyl phenol, 2-chlorophenol, 4-nitrophenol, pentachlorophenol, and phenol. The spikes are made up in methanol at 100 ug/mL for the base/neutrals and 200 ug/mL for the acids, as detailed in the appropriate extraction procedure. The volume spiked is 100 uL per 1.0 mL of extract, resulting in a concentration of 10 ng/uL for base/neutrals, and 20 ng/uL for acids.

NOTE: For all State of Wisconsin samples and when other projects require it, every analyte of interest must be spiked into the BS and MS/MSDs or only the BS, as applicable. All analytes must be spiked at 10 ng/uL before extraction.

13.0 SAMPLE PREPARATION

13.1 For sample extractions, refer to the appropriate procedures in section 5.0.

13.2 All samples must be spiked with surrogates before extracting (Refer to section 12.4).

13.3 All extracts must be spiked with internal standards, immediately prior to injection (Refer to section 12.2).

13.4 Extracts that are dark in color or thick and viscous will be diluted at least 1:10 prior to analysis.

14.0 CALIBRATION PROCEDURES

14.1 Set up instrumentation as specified in section 9.1.1 and 9.1.2.

14.2 All samples and standards are introduced into the GC/MS, by 1.0 uL direct injection. All injections are performed using an autosampler.

14.3 Each GC/MS system is hardware-tuned to meet Table 3 or Table 3A criteria for a 5.0 ng, 1.0 uL DFTPP injection. No analysis may begin until these criteria are met. Mass spectra are taken across the 198 ion apex. Background subtraction is designed only to eliminate column bleed or instrument background ions.

14.3.1 The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and/or DDD must not exceed 20%. Calculate degradation as follows:

$$\% \text{ degradation} = \frac{\text{Area}_{DDE} + \text{Area}_{DDD}}{\text{Area}_{DDE} + \text{Area}_{DDD} + \text{Area}_{DDT}} \times 100$$

14.3.2 The degradation result must be recorded in the daily instrument run logbook.

14.3.3 Pentachlorophenol and benzidine must be present at their normal responses, with minimal peak tailing. If peak tailing is suspect, calculate a tailing factor using the pentachlorophenol total ion chromatogram. To determine if corrective action is required, follow Figure 1 instructions. A tailing factor of 1 to 2 is considered acceptable, a tailing factor of 2 to 4 is considered the warning limit, and a tailing factor of 4 or higher is considered unacceptable, requiring corrective action. Corrective action for degradation and/or poor chromatography can include cleaning the injection port and clipping the first 6 to 12 cm off the capillary column.

14.4 All analytes must be quantified using the method of internal standards. Analytes and their corresponding internal standards are provided in Tables 1 and 1A. For analytes not listed, the corresponding internal standard must be the one eluting nearest the unlisted analyte. Primary quantitation ions are also provided in Tables 1 and 1A. A secondary ion may be used for quantitation, only if interferences are noted. If a secondary ion is used, the compound RF must be recalculated before quantitation.

14.5 Initial Calibration:

14.5.1 Calibration standards: The initial calibration for all analytes of interest must contain a minimum of five points (three for method 625). Table 1 standards calibration is typically run at 0.5, 1.0, 2.0, 5.0, 10, 15, 20, 25 ug/mL. The 25 ug/mL standard may be omitted. However, omitting lowers the linear calibration range. Each 1.0 mL calibration standard aliquot must be spiked with 5.0 uL of internal standard solution, immediately prior to analysis. Table 1A and other analytes, are run at the appropriate concentration, based on response. The lowest concentration of the initial calibration must be at the reporting limit.

14.5.2 Injections of 1.0 uL for each calibration standard, including surrogates and internal standards are analyzed. Figures 2 and 2A show chromatograms of a continuing calibration standard. A Response Factor (RF) is calculated for each compound using the Table 1 or 1A primary ions. Response factors are calculated as follows:

$$RF = \frac{A_X \times C_{IS}}{A_{IS} \times C_X}$$

where:

A_x = Area of the primary ion for the compound being measured
A_{is} = Area of the primary ion for the specific internal standard
C_x = Concentration of the compound being measured (ng/uL)
C_{is} = Concentration of the specific internal standard (ng/uL)

14.5.3 A system performance check must be performed to ensure that average RFs are acceptable for use in quantitation. System Performance Check Compounds (SPCCs) are listed in Table 4. The minimum acceptable average RF for SPCC compounds is 0.050. SPCCs typically have very low RFs (0.1to 0.2) and tend to decrease in response as the chromatographic system or the standards deteriorate. SPCCs are usually the first to show poor performance, and must meet the minimum requirement when the system is calibrated. The minimum recommended RF for all non-SPCC analytes of interest is 0.01, however, spectra quality and background (signal to noise) must also be considered.

14.5.4 Percent relative standard deviation (RSD) must be calculated for each compound using the following equation:

$$\%RSD = \frac{SD}{RF} \times 100$$

where:

SD = The standard deviation of the average response factor across the initial calibration curve.
RF = Average response factor across the initial calibration curve.

14.5.5 Percent RSD for each compound must be less than 15% to use an RF for quantitation, which assumes linearity through the origin. Percent RSD for each Table 5 calibration check compound (CCC) **must** be less than 30%. If percent RSD for any CCC is between 15% and 30%, a calibration curve must be constructed as outlined in 14.5.8. If percent RSD is **greater** than 30% for any CCC, the system is too unstable to proceed. Corrective action, including replacing the injection port liner, replacing or clipping the capillary column, and recalibration of the GC/MS system, must be initiated.

14.5.6 The relative retention time of each compound of interest in each calibration run must agree within 0.06 relative retention time units.

14.5.7 If percent RSD of any non-CCC compound is 15% or less, the response factor is assumed to be constant over the calibration range, and the average response factor can be used for quantitation.

14.5.8 If percent RSD for any compound is greater than 15%, a regression curve must be constructed by plotting area ratio ($Area_{analyte}/Area_{IS}$) against concentration. A first order or higher regression fit is used. The curve must not be forced through the origin:

14.5.8.1 Linear regression requires five calibration standards and is calculated as follows:

$$C_s = [((A_s * C_{is})/A_{is})-b]/a$$

Where:
A_s = Area of the target analyte peak, in sample "s"
A_{is} = Area of the "is" internal standard peak
C_s = Concentration of the target analyte in calibration standard "s"
C_{is} = Concentration of the internal standard "is"
a = Slope of the curve (also called the coefficient of C)
b = The intercept

14.5.8.2 A quadratic (second order) fit requires six calibration standards and a chromatographic curve fitting program, to solve the following:

$$[(A_s * C_{is})/A_{is}] = a * C_s^2 + b * C_s + c$$

Where:
c = the constant

14.5.8.3 A third order polynomial requires seven standards and a chromatographic curve fitting program, to solve the following:

$$[(A_s * C_{is})/A_{is}] = a * C_s^3 + b * C_s^2 + c * C_s + d$$

Where:
d = the constant

14.5.8.4 Analysts may select each analyte regression curve giving the least calibration error, as indicated by the correlation coefficient. To be acceptable, the correlation coefficient "r" for a first order regression must be 0.99 or higher. For second and third order regressions, the coefficient of determination (COD) "r^2" must be 0.99 or higher.

NOTE: Non-linear regression calibration must not be used as compensation for improper instrument maintenance. Non-linear regression for any analyte must not be employed on an instrument previously shown to **routinely** exhibit linearity.

14.5.9 If initial calibration criteria are not achieved using all calibration points run, the lowest or highest point on the curve may be dropped, provided enough points remain for the calibration model chosen.

14.5.9.1 A minimum of six calibration points are required to use second order regression curves. Five data points are needed for first order.

14.5.9.2 The lowest calibration point defines an analyte reporting limit. The low point can only be dropped provided required reporting limits are not affected.

14.5.9.3 Dropping the highest point shortens the calibration range, which could lead to a greater number of sample dilutions.

14.5.10 Run a second-source calibration standard containing all analytes at a mid-range concentration following the initial calibration. All analytes must have recoveries between 75-125% for the initial calibration to be considered valid. If these criteria are not met, analyze the second-source standard a second time. If the second analysis fails, take corrective action up to and including preparation and running of new initial calibration standard solutions. Repeat the second-source analysis for confirmation that corrective action was effective.

14.6 Continuing Calibration:

14.6.1 Prior to analysis of samples or standards, the DFTPP tuning standard is analyzed and evaluated, as specified in section 14.3. Acceptable DFTPP tuning must be demonstrated at the beginning of each 12-hour shift.

14.6.2 Immediately following every acceptable DFTPP analysis, a continuing calibration standard must be run at a mid-level concentration. For method 8270C analytes listed in Table 1, the concentration is 10 ug/mL. A continuing calibration standard contains all semi-volatile target analytes, including all surrogates and internal standards. Table 1A and other analytes, are run at the appropriate concentration, based on response. Each analyte RF is compared with the initial calibration average RF, from the same instrument.

14.6.3 System Performance Check Compounds (SPCC): A system performance check is made on every continuing calibration standard. All SPCC criteria must be met for a continuing calibration standard to pass. Table 4 lists SPCC compounds used for the continuing calibration. The minimum SPCC RF is 0.050. The recommended minimum RF for all non-SPCC compounds of interest is 0.01. However, spectra quality and background (signal to noise) must also be considered. If minimum response factors are not met for SPCCs, the system must be evaluated and corrective action taken, before analysis can continue. Possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front of the analytical column, and active sites in the column or chromatographic system.

14.6.4 Calibration Check Compounds (CCC): After the system performance check is met, Table 5 CCCs are used to check the initial calibration validity.

When average response factor is used for calibration, calculate percent difference between the initial calibration average RF and the continuing calibration RF:

$$\% \ Difference = \frac{RF_v - RF_m}{RF_m} \times 100$$

where:

RF_v = response factor from verification standard
RF_m = average calibration factor from initial calibration

Use percent drift to determine acceptance when calibrating using a regression fit model:

$$\% \ Drift = \frac{C_C - C_I}{C_I} \times 100$$

where:

C_I = theoretical CCC standard concentration
C_C = measured concentration using appropriate regression fit.

14.6.5 If percent difference or drift for every CCC is less than ±20%, the initial calibration remains valid. If any CCC compound differs from the initial calibration curve by greater than ±20%, corrective action must be taken. Problems similar to those listed under section 14.6.3 can also affect CCC results. If the problem source cannot be determined after corrective action has taken place, the calibration is considered out of control. At a minimum, the continuing calibration standard must be re-run. If necessary, a new calibration will be run. All CCC criteria must be met before sample analysis can continue. For non-CCC analytes, the percent difference or drift must be ≤25%, with the exception of poor performing analytes, which can have up to four outside of ≤25%, but must be ≤40%. Examples of poor performing analytes include: Chloro- and Nitro-anilines, Benzoic acid, Benzidine, Hexachlorocyclopentadiene, 4-Nitrophenol, and 2,4-Dinitrophenol. In addition, if response for any non-CCC compound is outside this criteria, but is recovering on the high side (response for the analyte is out of control high), then extracts with non-detect results for these compounds may still be used since there is no question of analyte sensitivity for the day. For method 625, all compounds must have a percent difference of less than ≤20%.

14.6.6 Internal standard responses and retention times in continuing calibration verification standards and samples must be evaluated in each run. If retention time for any internal standard varies by more than 30 seconds from the last calibration verification, the system must be inspected for malfunctions, and corrective action performed as necessary. If the EICP area for any internal standard varies by a factor of 2 (-50% to +100%) from the midpoint standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrective action performed as necessary. All sample extracts analyzed under either of these out of control conditions must be reanalyzed.

15.0 ANALYTICAL PROCEDURE

15.1 Every 12-hour shift **must** begin with the analysis of DFTPP, followed by a new calibration or a continuing calibration verification standard, then followed by an instrument blank.

15.2 The DFTPP tune **must** pass the tuning criteria in section 14.3 before subsequent analysis can begin. The 12-hour acquisition window **must** begin at the injection time of DFTPP. The entire 12-hour shift must be run under the same mass spectrometer conditions used for DFTPP. All samples must be run under the same gas chromatographic conditions as the initial calibration. Refer to sections 9.1.1 and 9.1.2 for recommended operating conditions.

15.3 Any calibrated compound **must** have an acceptable average Response Factor (RF) generated from the initial calibration curve, or be calibrated using a first or higher order regression curve. If Method 8270C is being utilized, the calibration **must** pass the SPCC and CCC criteria specified in section 14.5.

15.4 A continuing calibration standard is run on days an initial calibration is not. The continuing calibration standard **must** pass the criteria specified in section 14.6.

15.4.1 After the DFTPP and continuing calibration criteria are met, the analytical system **must** be demonstrated free from contamination by running an instrument blank before sample analysis during each 12-hour shift (Refer to section 18.3).

15.5 Once the above criteria are met, sample analysis may proceed for the remainder of the 12-hour shift.

15.6 If response for any analyte in the sample exceeds the initial calibration range, a dilution of the extract must be made. Attempt to dilute the sample only enough to bring the analyte(s) in question into the upper half of the calibration range. Additional internal standard must be added to the diluted extract to maintain the required 5.0 ng/uL internal standard concentration. The diluted extract is then reanalyzed.

15.7 A matrix spike, matrix spike duplicate, method preparation blank, and laboratory fortified blank **must** be run for each quality control batch to assess extraction efficiency. Up to a maximum of 20 samples/batch is allowed. It is preferred but not required that matrix quality control be analyzed with samples from the same extraction batch during one 12-hour shift. Additional quality control requirements are specified in section 18.0.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Qualitative analysis:

16.1.1 An analyte is identified by a mass spectra comparison between the sample and a standard of the suspected compound (standard reference spectrum). Standard reference spectra are obtained through analysis of the calibration. Two criteria must be satisfied to verify identification. First, a sample component must elute at the same GC relative retention time (RRT) as the standard compound. Secondly, the mass spectra of the sample component and standard compound must agree.

16.1.1.1 The sample component RRT must fall within ± 0.06 RRT units of the standard compound, in the daily continuing calibration standard. If interfering co-elution

prohibits accurate sample component RRT measurement from the total ion chromatogram, the RRT must be assigned by using extracted ion current profiles unique to the compound of interest.

16.1.1.2 All ions in standard mass spectra at a relative intensity greater than 10% are automatically checked by the software to be in a sample spectrum. The most abundant ion in standard mass spectra equals 100%. Relative ion intensities between standard and sample spectra, must agree within ± 30%. For example, an ion with 50% abundance in standard spectra, means the corresponding sample abundance must be between 20% and 80%.

16.1.2 For samples containing components not associated with calibration standards, a library search can be made for a tentative identification. Only after visual comparison of sample spectra with the nearest library searches must the analyst assign a tentative identification. When a tentatively identified compound (TIC) cannot be identified by name, a generic description must be given to identify functional groups. TIC nomenclature by functional group is as follows:

Name	**CAS #**
Unknown Alcohol	xx-xx-xx
Unknown Freon	xx-xxx-xxxx
Unknown Ketone	xx-xxx-xx
Unknown Acid	xxx-xx-xxxx
Unknown Hydrocarbon	xxx-xxx
Unknown Glycol Ethers	xxx-xx-x
Unknown Substituted Benzenes	xxxxx
Hydrocarbons (sub. Benzenes)	xxxxx-x-x
Hydrocarbons, Total	xxxxxx

Guidelines for making tentative identification are:

16.1.2.1 Relative intensities of major ions in a reference spectrum (ions with >10% of the most abundant ion) should be present in the sample spectrum.

16.1.2.2 Relative intensities of major ions should agree within ± 20%. For example, an ion with 50% abundance in a standard spectrum corresponds to a sample ion abundance between 30% and 70%.

16.1.2.3 Molecular ions in the reference spectrum should be present in the sample spectrum.

16.1.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for background contamination or co-eluting components.

16.1.2.5 Ions in the reference spectrum but not in the sample spectrum should be reviewed for subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create such discrepancies. TIC peaks are determined by the following search criteria:

16.1.2.5.1 The peak in question must be >10% of the nearest internal standard.

16.1.2.5.2 The top 20 potential TICs must be reviewed, unless otherwise required. The match quality must be 70% or higher to report a positive identification, in the absence of interfering peaks.

NOTE: A 70% match corresponds to a fit of 700 on the Ion Trap or a Q-value of 70 on the MSD. If there are interfering peaks, analyst discretion must be employed when reporting matches.

16.2 Quantitative analysis:

16.2.1 When a compound has been identified, quantitation is performed using area of the appropriate quantitation ion(s), listed in Tables 1 and 1A. The internal standards technique is used. Internal standards are specified in Tables 1 and 1A. For target analytes in neither table, the internal standard nearest the analyte retention time of the analyte must be used. All qualitative results must be reported without correction for recovery, and without blank subtraction.

16.2.2 Calculate analyte concentration as follows:

Water:

$$\text{Concentration in ug/L} = \frac{(A_x)(I_s)(V_t)(\text{Dil})}{(A_{is})(\text{RF})(V_o)}$$

where:

A_x = Characteristic ion area, for the compound being quantitated
I_s = Internal standard concentration injected, in ug/mL
V_t = Total extract volume, in mL
A_{is} = Internal standard characteristic ion area
RF = Average response factor, for compound being quantitated (section 14.5.2)
V_o = Sample volume extracted, in liters
Dil = Any post extraction dilution factor

Sediment/Soil Sludge:

$$\text{Concentration in mg/kg} = \frac{(A_x)(I_s)(V_t)(\text{Dil})}{(A_{is})(\text{RF})(W_s)(D)}$$

where:

W_s = Sample mass extracted, in grams
D = Sample % solids, as a decimal fraction

All other variables are the same..

NOTE: If any analyte initial calibration RF has not met the 15% RSD requirement, a regression curve must be used to determine extract concentration and the calculations modified accordingly.

16.2.3 When requested, the concentration of non-calibrated sample components must be estimated. The formulas above are used with the following modifications:

16.2.3.1 Areas A_x and A_{is} are from total ion chromatograms, and the compound RF is assumed to be 1.

16.2.3.2 Use the nearest interference-free internal standard.

16.2.3.3 The concentration reported must be qualified as estimated.

16.3 Manual Integrations

16.3.1 The TriMatrix policy concerning manual integration must be followed without exception. Every effort must be made to set up data processing software integration parameters, to avoid manual integration.

16.3.2 All manual integration must be documented by printing both the original integration performed by the data system, and the manually integrated ion chromatograms. Manual integrations must be initialed and dated, and must have the rationale for the new integration clearly stated on the print out. These must be included in all client package deliverables.

16.3.3 When required, manual integration must be consistently applied to standards and samples. Examples where manual integration may be necessary include: closely eluting isomers (1,3- and 1,4-Dichlorobezene), jagged analyte peak shapes split by the integrator, and analytes like benzidine that have significant peak-tailing.

16.3.4 **Under no circumstances must manual integration be applied solely for the purpose of meeting quality control. Peak shaving and peak enhancing are strictly prohibited.**

16.3.5 For further details, refer to TriMatrix Laboratories SOP GR-10-115, *Manual Integrations*, latest revision.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are also responsible for correctly filling in, turning in, and filing the associated paperwork. It is essential to perform these tasks, to provide defensible data to clients.

17.2 ELEMENT Reporting

17.2.1 When an analysis sequence is completed, all data must be uploaded to the ELEMENT DataSystem. For details on how to upload data, refer to TriMatrix SOP GR-10-123. Carefully

review the *data entry* table to insure that results are accurately reported and associated with the correct quality control batch. Manually enter a dilution factor for each corresponding sample. Dilution factors are used to calculate elevated reporting limits from diluting an extract. The dilution factor is a multiplier that may or may not reflect actual dilution volumes.

17.2.2 If internal chain-of-custody is required, it is important that a COC form be filled in correctly, and returned to the COC file location.

17.3 Laboratory Required Paperwork

17.3.1 All run and maintenance logbooks must be filled in completely and correctly. Corrections must be made with a lineout, not a write-over, and must be dated and initialized. Blank lines in run logbooks must be Z'd out. Refer to Figures 3 and 4 for standards forms and run logbook examples.

17.3.2 All tune, calibration, and continuing calibration standard runs must be placed in the correct file.

17.3.3 All ELEMENT documentation and raw data must be placed in the correct client folder and given to a data management technician. The technician must record the date and contents of the data then archive the folder.

17.4 CLP-like Deliverables

17.4.1 Include the following in data packages requiring CLP-like deliverables:

17.4.1.1 Copies of tunes, including the EICP. These copies must contain ion peaks for DFTPP, Benzidine, and Pentachlorophenol with tailing factor calculations and results, ion abundance results and requirements including mass listings, and associated Form Vs.

17.4.1.2 Copies of calibrations, including raw data quantitation reports, chromatograms, and associated Form VIs.

17.4.1.3 Copies of continuing calibration standards, including raw data quantitation reports, chromatograms, and associated Form VIIs.

17.4.1.4 Copies of raw sample data, including applicable quality control, quantitation reports, triple plots for positive results, chromatograms, and TIC reports when requested

17.4.1.5 Completed MPB summary Form IVs.

17.4.1.6 Completed Internal Standard recovery Form VIIIs.

17.4.1.7 Copies of supporting information, including: internal chain of custody forms, run and standards logbooks, method detection limits, and extraction summaries.

NOTE: Forms I, II, and III are generated from ELEMENT and must be included with the deliverables package.

18.0 QUALITY ASSURANCE

18.1 Quality control requirements are included in the procedure, as follows:

18.1.1 DFTPP tune (section 14.3)

18.1.2 Initial Calibration (section 14.5)

18.1.3 Continuing Calibration (section 14.6)

18.1.4 In addition, quality control must include sections 18.3 through 18.7:

18.2 Blanks

18.2.1 After the DFTPP tune and calibration or continuing calibration have been run, a blank is required before sample analysis showing that the system is free from interferences and contamination at or below the reporting limit. The blank is a Method Preparation Blank (BLK) or a solvent instrument blank (IBL). An MPB is an extracted blank from a sample batch. An IBL is methylene chloride spiked only with internal standard. A BLK may be used for the blank if uncontaminated. Otherwise an IBL must be run. Run a BLK or IBL at least once each 12-hour shift. Run blanks more frequently if carryover is suspected from a high-concentration sample, or if laboratory contamination is in question. Extract a BLK for each batch of up to 20 samples. BLKs must be spiked with surrogates and carried through the same preparation as samples. Acceptance criteria for both the IBL and BLK are that all target analytes must have concentrations below the lowest reporting limit for samples analyzed that day. Common laboratory contaminants such as phthalates must have blank concentrations less than five times the reporting limit.

18.3 Internal Standards

18.3.1 Internal standard responses and retention times in all runs following the continuing calibration standard must be evaluated during or immediately after data acquisition. All internal standard retention times must be within ± 30 seconds of the current 12-hour continuing calibration standard. All internal standard quantitation ion areas must stay within a factor of two (-50% to +100%) of the mid-point standard from the initial calibration.

18.3.2 If at any time an internal standard fails the -50% to +100% area window, the ability to accurately quantitate analytes is reduced. Every effort must be made to prevent an internal standard failure, including dilution and reanalysis of samples. Refer to TriMatrix GR-04-101 for the corrective action sequence, should internal standards fail. If many samples are out-of-control for no apparent reason, the mass spectrometer must be inspected for a malfunction and appropriate repairs made. Once the problem is resolved, samples analyzed with the faulty spectrometer must be re-analyzed, with acceptable internal standard responses.

18.4 Surrogates

18.4.1 All samples must be spiked with surrogates. Until twenty samples of a given matrix have been analyzed, a default recovery window of 50% - 150% will be used. When twenty samples of a given matrix have been analyzed, in-house recovery limits will be generated (section 18.5.2-18.5.4) and used. Table 6 provides examples of in-house surrogate recovery limits. These are examples only. At a minimum, surrogate recovery limits must be updated annually, on a matrix-by-matrix basis.

18.4.2 Calculate upper and lower control limits for each surrogate, as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

where
p = average percent recovery
s = standard deviation of the average

18.4.3 Two standard deviations will be used when three give a negative lower control limit.

18.4.4 If surrogate recovery is not within limits, take corrective action. Refer to TriMatrix GR-04-101 for the corrective action sequence, including re-extraction when sample volumes and holding times permit, and data qualification. If many surrogates are out-of-control for no apparent reason, the instrument must be inspected for a malfunction, and appropriate repairs made. When the problem is resolved, samples analyzed with the faulty instrument must be reanalyzed with acceptable surrogate recovery.

18.5 Matrix Spikes and Laboratory Fortified Blanks

18.5.1 To assess extraction efficiency, a Matrix Spike (MS), Matrix Spike Duplicate (MSD), and Blank Spike (BS) must be extracted. Spiking must contain the compounds in Table 7 and must be performed at least once every 20 samples for each matrix extracted. If only one to ten samples are analyzed in a month, at least one matrix spike, one matrix spike duplicate and one laboratory fortified blank is required. A BS must be extracted daily or with each sample batch, whichever is more frequent. Generally, the MPB and BS are run at the beginning of the 12-hour shift, and matrix spikes at the end. Running matrix spikes last helps document that the instrument is still operating correctly toward the end of the 12-hour shift.

NOTE: For State of Wisconsin samples and where project specifications require, all target analytes must be added for associated spikes, spike duplicates and BSs.

18.5.2 Until five matrix spikes, matrix spike duplicates, and laboratory fortified blanks have been analyzed, recoveries must be validated against default recovery limits of 50% - 150%. Matrix Spike/Matrix Spike Duplicate relative percent difference (RPD) limits must use a default maximum limit of 20%.

18.5.3 When five data sets of a given matrix (water, soil, other) have been analyzed, calculate average percent recovery (R) and standard deviation of the average (SD). Express the recovery as the percent recovery interval from R ± 3SD. For example, if R = 90% and SD = 10%, spike recovery limits are 60% - 120%. Separate limits must be calculated for laboratory fortified blanks. The relative percent difference limit must be calculated by averaging five MS/MSD percent differences ± 3 standard deviations. Acceptance limits will be updated at least annually.

18.5.4 Calculate percent recovery as follows:

$$\%\,\text{recovery} = \frac{(A_{spk} - A_{smp})}{SPK} \times 100$$

where:
A_{spk} = The spiked sample concentration, in ug/L or mg/kg
A_{smp} = The non-spiked sample concentration, in ug/L or mg/kg

$$SPK = \left(\frac{\text{concentration of spiked standard in ug/L x mL spiked}}{\text{initial sample volume in L}} \right)$$

18.5.5 Calculate relative percent difference (RPD) as follows:

$$\%RPD = \frac{|(MS - MSD)|}{\left[\frac{(MS + MSD)}{2}\right]} \times 100$$

where:

MS = The matrix spike concentration, in ug/L or mg/kg
MSD = The matrix spike duplicate concentration, in ug/L or mg/kg

18.5.6 If recovery or duplication are not within limits, refer to TriMatrix GR-04-101 for the corrective action sequence, to determine if or how data is qualified.

18.5.7 If a BS analyte is out-of-control, the problem must be immediately identified and corrected. Samples for the analyte must not be run until the problem is corrected. The purpose of a BS is to verify that out-of-control compounds in a MS or MSD are the result of matrix interference, rather than extraction or system error. Failure of any analyte in a BS, requires corrective action specific to the failed parameter. Every effort must be made to determine the cause for failure (mis-spiked, mis-extracted, or any other reason). When the problem is located, corrective action must be taken following TriMatrix SOP GR-04-101. If it is determined that re-extraction is necessary, the entire batch of samples must be re-extracted.

NOTE: If a BS recovery is biased high and there are no positive results for the analyte in the associated samples, results can be reported without the need for corrective action or qualification.

18.6 Method Detection Limit Studies

18.6.1 A Method Detection Limit (MDL) study must be performed annually for every target compound analyzed. MDL studies must be performed on each instrument involved in sample analysis. Analyte results for which a study has not been performed must be reported as estimated. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero. Actual reporting limits are derived from the MDL study. Minimum reporting limits are equal to the concentration spiked for the MDL study, provided the MDL passes. Reporting limits actually achieved for any given analysis will vary depending on instrument sensitivity, matrix, and extract dilutions. MDL studies must be run for aqueous and soil matrices.

18.6.2 The procedure followed for MDL studies is based on the method given in 40 Code of Federal Regulations, Part 136, Appendix B, latest revision. Method 8270C quality control governing the analysis must be followed.

18.6.3 Seven replicate extractions and subsequent analyses are performed using reagent water or Ottawa Sand spiked with the target compounds of interest at estimated minimum reporting limit concentrations.

18.6.4 For each target compound, standard deviation of the average is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

18.6.5 If the concentration spiked is greater than or equal to the calculated MDL, and less than or equal to five times the calculated MDL, and there are no zero percent recoveries in the seven results, the MDL is acceptable. The MDL study must be repeated, only for the analyte(s) that did not pass. If a study needs repeated at a different concentration, the entire set of seven needs to be repeated. If a study does not pass due to poor reproducibility on one result, only that result needs repeated. Only one result may be rejected and repeated however. All seven results do not need to be obtained during the same 12-hour shift.

18.6.6 If a reporting limit is above a client or state-desired reporting limit, the calculated MDL may be used provided results are narrated. Narration must state that the reporting limit is the calculated MDL and when spiked at that level, analyte was not observed.

18.6.7 Table 6 is an MDL spreadsheet example. The spreadsheet is used to calculate and verify MDLs and reporting limits.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a one time demonstration of capability study. While the demonstration of capability is not instrument dependent, a study is required on every instrument running samples, to demonstrate each instrument's ability to generate acceptable accuracy and precision.

19.2 Prepare a 100 ug/L concentration of all compounds of interest in four 1000 mL aliquots of reagent water, by spiking with 0.100 mL of a 100 ng/uL standard. Extract to a 10 mL final volume. The spiking solution must be prepared independently from stock standards used for quantitation.

19.3 Analyze the four extracts using Method 8270C quality control, and following every step of the procedure. For each target compound, calculate average percent recovery "x" and standard deviation "s" in ug/L. Use all four results. Calculate by inputting into the IDC spreadsheet located on the laboratory intranet subdirectory "\\library\spreadsheets\training documents\qc_idc-demonstration of capability(doc).xls".

19.3.1 For each target compound, "x" must pass the current ELEMENT BS acceptance limits. Also, relative standard deviation must be less than or equal to 20%. Calculate %RSD as [("s"/"x")*100]. If "s" and %RSD for all analytes pass both acceptance criteria, the demonstration of capability study is complete. The analyst and associated instrument may run samples. If %RSD exceeds 20% or "x" falls outside the current ELEMENT BS acceptance limits, analyst or instrument performance is unacceptable for the analyte.

19.3.2 When one or more analytes fail for "x" or %RSD, proceed according to section 19.4.

19.4 Locate and correct the source of any problem and repeat the study for failed analytes. Repeated failure however, will confirm a general problem with the procedure or technique used. If this occurs, locate and correct the problem, then repeat the study for all unacceptable compounds beginning with section 19.1. Samples may not be analyzed by any analyst or on any instrument, until a demonstration of capability study has been successfully completed. Copies of successful demonstration of capability studies, IDC spreadsheets and raw data must be given to Quality Assurance.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all procedures in TriMatrix Laboratory SOP number GR-15-102, *Laboratory Waste Disposal*, most recent revision, for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 3, December 1996

22.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids

22.3 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. B, Definition and Procedure for the Determination of the Method Detection Limit

23.0 ATTACHMENTS

23.1	Table 1	Routine Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions
23.2	Table 1A	Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions
23.3	Table 2	Default Reporting Limits
23.4	Table 2A	Default Reporting Limits
23.5	Table 3	DFTPP Key Ions and Ion Abundance Criteria (EPA CLP)
	Table 3A	DFTPP Key Ions and Ion Abundance Criteria (Method 8270C Default Criteria)
23.6	Table 4	System Performance Check Compounds (SPCC)
23.7	Table 5	Continuing Check Compounds (CCC)
23.8	Table 6	Method Detection Limit Study Example (Partial Table)
23.9	Table 7	Demonstration of Capability Study Example (Partial Table)
23.10	Figure 1	Pentachlorophenol Tailing Factor
23.11	Figure 2	Continuing Calibration Standard Total Ion Chromatogram, Varian Ion Trap (10 ug/mL)
23.12	Figure 2A	Continuing Calibration Standard Total Ion Chromatogram, Agilent 5973 MSD (10 ug/mL)
23.13	Figure 3	Standards Form Example
23.14	Figure 4	Analytical Run Logbook Example

Approved By: _____ 1-22-09 **QA Officer** **Approved By:** _____ 1/23/09 **Area Supervisor**

23.15 Method 8270C Selected Ion Monitoring (SIM) instructions

Attachment 23.1 (Table 1)
Routine Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
Acenaphthene-d_{10} (I.S.)	17.76	162	164, 160	3
Chrysene-d_{12} (I.S.)	25.04	240	120, 236	5
Dichlorobenzene-1,4-d_4 (I.S.)	10.06	152	150, 115	1
Naphthalene-d_8 (I.S.)	13.21	136	68	2
Perylene-d_{12} (I.S.)	29.83	264+265 (264)**	260, 265	6
Phenanthrene-d_{10} (I.S.)	20.50	188	94,80	4
2-Fluorobiphenyl (surr.)	16.00	172	171	3
2-Fluorophenol (surr.)	7.18	112	64	1
Nitrobenzene-d_5 (surr.)	11.38	127+128 (128)**	82, 54	2
Phenol-d_6 (surr.)	9.13	99		1
2,4,6-Tribromophenol (surr.)	19.33	330	332, 141	3
Terphenyl-o (surr.)	21.03	230	215,229	5
Acenaphthene	17.84	152+154 (154)**	153, 152	3
Acenaphthylene	17.44	152	151, 153	3
Anthracene	20.63	178	176, 179	4
Benzoic acid	12.39	105	77,122	2
Benzo(a)anthracene	25.00	228	229, 226	5
Benzo(b and k)fluoranthene	28.24/28.40	252	253, 125	6
Benzo(g,h,i)perylene	37.32	276+277 (276)**	138, 277	6
Benzo(a)pyrene	29.60	252	253, 125	6
Benzyl alcohol	10.35	108	79, 77	1
Bis(2-chloroethoxy)methane	12.51	92+93 (93)**	95, 123	2
Bis(2-chloroethyl)ether	9.38	92+93 (93)**	63, 95	1
Bis(2-chloroisopropyl)ether	10.65	45	77, 121	1
Bis(2-ethylhexyl)phthalate	24.59	149	167, 279	5
4-Bromophenyl phenyl ether	19.73	248	250, 141	4
Butyl benzyl phthalate	23.56	149	91, 206	5
4-Chloroaniline	13.40	127	129	2
2-Chloronaphthalene	16.34	162	127, 164	3
4-Chloro-3-methylphenol	14.60	107	144, 142	2
2-Chlorophenol	9.57	128	64, 130	1
4-Chlorophenyl phenyl ether	18.87	204	206, 141	3

Approved By: _____ 1-22-09 **QA Officer**

Approved By: _____ 1/23/09 **Area Supervisor**

Attachment 23.1 (Table 1) - Continued
Routine Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
Chrysene	25.10	228	226, 229	5
Dibenz(a,h)anthracene	35.57	278	139, 279	6
Dibenzofuran	18.20	168	139	3
Di-n-butylphthalate	21.23	149	150, 104	4
1,3-Dichlorobenzene	9.93	146	148, 111	1
1,4-Dichlorobenzene	10.10	146	148, 111	1
1,2-Dichlorobenzene	10.45	146	148, 111	1
3,3'-Dichlorobenzidine	24.80	252	254, 126	5
2,4-Dichlorophenol	12.84	162	164, 98	2
Diethylphthalate	18.59	149+177 (149)**	177, 150	3
2,4-Dimethylphenol	12.26	107	121, 122	2
Dimethylphthalate	17.03	163	194, 164	3
4,6-Dinitro-2-methylphenol	18.94	198	51, 105	4
2,4-Dinitrophenol	17.86	184	63, 154	3
2,4-Dinitrotoluene	18.12	165	63, 89	3
2,6-Dinitrotoluene	17.20	165	63, 89	3
1,2-Diphenylhydrazine	19.15	77	105, 182	4
Di-n-octylphthalate	26.32	149	167, 43	5
Fluoranthene	22.35	202	101, 203	4
Fluorene	18.89	166	165, 167	3
Hexachlorobenzene	19.87	284	142, 249	4
Hexachlorobutadiene	13.54	225	223, 227	2
Hexachlorocyclopentadiene	15.45	237	235, 272	3
Hexachloroethane	11.30	119	201, 199	1
Indeno(1,2,3-cd)pyrene	35.52	276+277 (276)**	138, 227	5
Isophorone	12.00	82	95, 138	2
2-Methylnapthalene	15.06	142	141	2
2-Methylphenol (o-cresol)	10.58	108	79,107	1
3- and/or 4-Methylphenol (m,p-cresol)	10.95	108	79,107	1
Naphthalene	13.26	128	129, 127	2

Attachment 23.1 (Table 1) - Continued
Routine Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
2-Nitroaniline	16.59	138	92, 138	3
3-Nitroaniline	17.62	138	108, 92	3
4-Nitroaniline	18.90	108+138 (138)**	108, 92	3
Nitrobenzene	11.43	77+123 (77)**	65	2
2-Nitrophenol	12.21	109+139 (139) **	109, 65	2
4-Nitrophenol	18.02	65	109, 65	3
N-Nitrosodimethylamine	4.58	73+74 (74)**	42	1
N-Nitrosodiphenylamine	19.08	169	168, 167	4
N-Nitrosodipropylamine	10.95	70	42, 101, 1	1
Pentachlorophenol	20.18	266	264, 268	4
Phenanthrene	20.54	178	179, 176	4
Phenol	9.17	94	65, 66	1
Pyrene	22.74	202	200, 203	5
1,2,4-Trichlorobenzene	13.05	180	182, 145	2
2,4,5-Trichlorophenol	15.91	196	198, 200	3
2,4,6-Trichlorophenol	15.79	196	198, 200	3

I.S. = Internal Standard
surr. = Surrogate
* retention time provided for example only.
** for MSD

Attachment 23.2 (Table 1A)
Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
Acenaphthene-d_{10} (I.S.)	17.05	162	160,164	3
Chrysene-d_{12} (I.S.)	24.14	240	120,236	5
Dichlorobenzene-1,4-d_4 (I.S.)	9.39	152	150,115	1
Naphthalene-d_8 (I.S.)	12.49	136	68	2
Perylene-d_{12} (I.S.)	28.22	264+265 (264)***	260	6
Phenanthrene-d_{10} (I.S.)	19.96	188	94,80	4
Acetophenone	10.45	105	77,70	2
2-Acetylaminofluorene	23.74	181	223,180	3
4-Aminobiphenyl	18.63	169	168,167	3
Aniline	8.76	93	66,65	1
Aramite**	22.64/22.73/22.85	185	63,57	4
2-sec-Butyl-4,6-dinitrophenol	19.89	211	163,147	3
2,6-Dichlorophenol	12.69	162	164,63	2
p-(Dimethylamino)azobenzene	22.71	120	225,77	3
7,12-Dimethylbenz(a)anthracene	26.79	256	252,241	5
3,3'-Dimethylbenzidine	23.26	212	213,106	3
2,2-Dimethylphenethylamine	12.64	58	91	2
1,3-Dinitrobenzene	16.55	76	139,169,50, 168	2
Carbazole	17.97	167	83, 139	4
Ethyl methacrylate	4.98	69	41,99	1
Ethyl methanesulfonate	7.74	79	109,97	1
Hexachlorophene	27.48	196	198,209	6
Hexachloropropene	12.75	213	215,211	2
Isosafrole	15.58	162	131,104	2
Methapyrilene	21.39	58	97,72	3
3-Methylcholanthrene	29.28	268	126,252	5
Methyl methacrylate	3.67	69	41,100	1
Methyl methanesulfonate	6.34	80	65,79	1
1,4-Naphthoquinone	16.23	158	104,102	2
1-Naphthylamine	17.87	143	115,116	2
2-Naphthylamine	18.05	143	115,116	2
4-Nitroquinoline-1-oxide	21.38	190	89,101	4

Approved By: _____ 1-22-09 **QA Officer** **Approved By:** _____ 1/23/09 **Area Supervisor**

Attachment 23.2 (Table 1A) - Continued
Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
N-Nitrosodi-n-butylamine	13.64	84	57,41	2
N-Nitrosodiethylamine	7.14	102	42,44	1
N-Nitrosomethylethylamine	5.75	88	42,43	1
N-Nitrosomorpholine	10.48	56	116,86	2
N-Nitrosopiperidine	11.24	114	42,56	2
N-Nitrosopyrrolidine	10.39	100	41,42	1
5-Nitro-o-toluidine	18.42	152	106,77	3
Pentachlorobenzene	17.46	250	252,248	3
Pentachloroethane	8.74	167	93,117	2
Pentachloronitrobenzene	19.78	237	169,142	4
Phenacetin	19.17	108	109,179	3
p-Phenylenediamine	13.80	108	80,107	2
2-Picoline	6.73	93	66,92	1
Pronamide	20.20	173	175,145	3
Pyridine	6.32	79	52,51	1
Safrole	14.24	162	131,101	2
1,2,4,5-Tetrachlorobenzene	14.76	216	214,218	3
2,3,4,6-Tetrachlorophenol	17.83	232	230,234	3
o-Toluidine	10.54	107	106,79	2
sym-Trinitrobenzene	19.09	75	74,213	3
Octachlorocyclopentene (C-58)	19.03	307+309	237	3

I.S. = Internal Standard
* retention time provided for example only
** Aramite chromatographs into three peaks
*** For MSD

Attachment 23.3 (Table 2)
Default Reporting Limits

Semivolatiles	CAS Number	Default Reporting Limits** Surface/Ground/ Wastewater ug/L	Low Soil/ Sediment mg/kg
Acenaphthene	83-32-9	5	0.33
Acenaphthylene	208-96-8	5	0.33
Anthracene	120-12-7	5	0.33
Benzoic acid	65-85-0	50	3.3
Benzo(a)anthracene	56-55-3	5	0.33
Benzo(b and k)fluoranthene	205-99-2 / 207-08-9	5	0.33
Benzo(g,h,i)perylene	191-24-2	5	0.33
Benzo(a)pyrene	50-32-8	5	0.33
Benzyl alcohol	100-51-6	50	1.3
Bis(2-chloroethoxy)methane	111-91-1	5	0.33
Bis(2-chloroethyl)ether	111-44-4	5	0.33
Bis(2-chloroisopropyl)ether	39638-32-9	5	0.33
Bis(2-ethylhexyl)phthalate	117-81-7	5	0.33
4-Bromophenyl phenyl ether	101-55-3	5	0.33
Butyl benzyl phthalate	85-68-7	5	0.33
Carbazole	86-74-8	5	0.33
4-Chloroaniline	106-47-8	20	1.3
2-Chloronaphthalene	91-58-7	5	0.33
4-Chloro-3-methylphenol	59-50-7	5	0.33
2-Chlorophenol	95-57-8	5	0.33
4-Chlorophenyl phenyl ether	7005-72-3	5	0.33
Chrysene	218-01-9	5	0.33
Dibenz(a,h)anthracene	53-70-3	5	0.33
Dibenzofuran	132-64-9	5	0.33
Di-n-butylphthalate	84-74-2	5	0.33
1,3-Dichlorobenzene	541-73-1	5	0.33
1,4-Dichlorobenzene	106-46-7	5	0.33
1,2-Dichlorobenzene	95-50-1	5	0.33
3,3'-Dichlorobenzidine	91-94-1	20	2.0
2,4-Dichlorophenol	120-83-2	5	0.33
Diethylphthalate	84-66-2	5	0.33

Approved By: _____ 1-22-09 **QA Officer** **Approved By:** _____ 1/23/09 **Area Supervisor**

Attachment 23.3 (Table 2) - Continued
Default Reporting Limits

Semivolatiles	CAS Number	Default Reporting Limits** Surface/Ground/ Wastewater ug/L	Low Soil/ Sediment mg/kg
2,4-Dimethylphenol	105-67-9	5	0.33
Dimethylphthalate	131-11-3	5	0.33
4,6-Dinitro-2-methylphenol	534-52-1	20	1.7
2,4-Dinitrophenol	51-28-5	20	1.7
2,4-Dinitrotoluene	121-14-2	5	0.33
2,6-Dinitrotoluene	606-20-2	5	0.33
Diphenylamine	122-39-4	5	0.33
Di-n-octylphthalate	117-84-0	5	0.33
Fluoranthene	206-44-0	5	0.33
Fluorene	86-73-7	5	0.33
Hexachlorobenzene	118-74-1	5	0.33
Hexachlorobutadiene	87-68-3	5	0.33
Hexachlorocyclopentadiene	77-47-4	5	0.33
Hexachloroethane	67-72-1	5	0.33
Indeno(1,2,3-cd)pyrene	193-39-5	5	0.33
Isophorone	78-59-1	5	0.33
2-Methylnaphthalene	91-57-6	5	0.33
2-Methylphenol (o-cresol)	95-48-7	5	0.33
3- and/or 4-Methylphenol (m,p-cresol)	106-44-5	5	0.33
Naphthalene	91-20-3	5	0.33
2-Nitroaniline	88-74-4	20	1.7
3-Nitroaniline	99-09-2	20	1.7
4-Nitroaniline	100-01-6	20	1.7
Nitrobenzene	98-95-3	5	0.33
2-Nitrophenol	88-75-5	5	0.33
4-Nitrophenol	100-02-7	20	1.7
N-Nitrosodimethylamine	62-75-9	5	0.33
N-Nitrosodiphenylamine	86-30-6	5	0.33
N-Nitrosodipropylamine	621-64-7	5	0.33
Pentachlorophenol	87-86-5	20	1.7

Attachment 23.3 (Table 2) - Continued
Default Reporting Limits

Semivolatiles	CAS Number	Default Reporting Limits** Surface/Ground/ Wastewater ug/L	Low Soil/ Sediment mg/kg
Phenanthrene	85-01-8	5	0.33
Phenol	108-95-2	5	0.33
Pyrene	129-00-0	5	0.33
1,2,4-Trichlorobenzene	120-82-1	5	0.33
2,4,5-Trichlorophenol	95-95-4	5	0.33
2,4,6-Trichlorophenol	88-06-2	5	0.33

* Sample reporting limits are highly matrix-dependent. The reporting limits listed are provided for guidance and may not always be achievable.

** Water reporting limits are based on an extraction of 1000 mL to 10 mL, in the MDL study. Reporting limits listed for soil/sediment are based on an extraction of 30 g to 10 mL.

Attachment 23.4 (Table 2A)
Additional Default Reporting Limits

Semivolatiles	CAS Number	Default Reporting Limits** Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
Acetophenone	98-86-2	10	0.33
2-Acetylaminofluorene	53-96-3	50	2.0
4-Aminobiphenyl	92-67-1	10	0.33
Aniline	62-53-3	5	1.7
Aramite	140-57-8	50	2.0
2-sec-Butyl-4,6-dinitrophenol	88-85-7	5	2.0
2,6-Dichlorophenol	87-65-0	5	0.33
p-(Dimethylamino)azobenzene	60-11-7	10	0.33
7,12-Dimethylbenz(a)anthracene	57-97-6	10	0.33
3,3'-Dimethylbenzidine	119-93-7	50	2.0
2,2-Dimethylphenethylamine	122-09-8	20	0.70
1,3-Dinitrobenzene	99-65-0	5	0.33
Ethyl methacrylate	97-63-2	50	2.0
Ethyl methanesulfonate	62-50-0	10	1.0
Hexachlorophene	70-30-4	***	***
Hexachloropropene	1888-71-7	50	2.0
Isosafrole	120-58-1	20	0.70
Methapyrilene	91-80-5	10	1.0
3-Methylcholanthrene	56-49-5	50	2.0
Methyl methacrylate	80-62-6	50	2.0
Methyl methanesulfonate	66-27-3	50	2.0
1,4-Naphthoquinone	130-15-4	1000	30
1-Naphthylamine	134-32-7	50	2.0
2-Naphthylamine	91-59-8	50	2.0
4-Nitroquinoline-1-oxide	56-57-5	500	20
N-Nitrosodi-n-butylamine	924-16-3	20	0.90
N-Nitrosodiethylamine	55-18-5	5	2.0
N-Nitrosomethylethylamine	62-75-9	5	2.0
N-Nitrosomorpholine	59-89-2	20	0.70
N-Nitrosopiperidine	100-75-4	20	0.70

Attachment 23.4 (Table 2A) - Continued
Additional Default Reporting Limits

Semivolatiles	CAS Number	Default Reporting Limits** Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
N-Nitrosopyrrolidine	930-55-2	20	0.70
5-Nitro-o-toluidine	99-55-8	10	0.33
Pentachlorobenzene	608-93-5	5	0.33
Pentachloroethane	76-01-7	50	2.0
Pentachloronitrobenzene	82-68-8	20	0.50
Phenacetin	62-44-2	10	0.33
p-Phenylenediamine	106-50-3	***	***
2-Picoline	109-06-8	20	0.70
Pronamide	23950-58-5	10	0.33
Pyridine	110-86-1	10	0.33
Safrole	94-59-7	10	0.33
1,2,4,5-Tetrachlorobenzene	95-94-3	10	0.50
2,3,4,6-Tetrachlorophenol	58-90-2	50	2.0
o-Toluidine	95-53-4	10	0.33
sym-Trinitrobenzene	99-35-4	20	0.70
Octachlorocyclopentene (C-58)	706-78-5	0.1	0.02

* Sample reporting limits are highly matrix-dependent. The listed values are provided for guidance and may not always be achievable.

** Water reporting limits are based on extracting 1000 mL to 1.0 mL, in the MDL study. Reporting limits listed for soil/sediment are based on extracting 30 g to 1.0 mL.

*** These compounds are demonstrated to be difficult to extract from water, or difficult to chromatograph. Method detection limits are not available.

Attachment 23.5 (Table 3)
DFTPP Key Ions and Ion Abundance Criteria
(EPA CLP)

Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	<2% of mass 69
69	Present
70	<2% of mass 69
127	25-75% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance (see note)
199	5-9% of mass 198
275	10-30% of mass 198
365	>0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15-24% of mass 442

NOTE: All ion abundances MUST be normalized to m/z 198 which is the nominal base peak, even though ion abundances of m/z 442 may be up to 110% that of m/z 198.

Attachment 23.5 (Table 3A)
DFTPP Key Ions and Ion Abundance Criteria
(Method 8270C Defaults)

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Approved By: _____ 1-22-09 **Approved By:** _____ 1/23/09
QA Officer **Area Supervisor**

Attachment 23.6 (Table 4)
System Performance Check Compounds (SPCC)

Base/Neutral Fraction	Acid Fraction
N-Nitrosodi-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

Attachment 23.7 (Table 5)
Calibration Check Compounds (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Attachment 23.8 (Table 6)
Method Detection Limit Study Example

TriMatrix Laboratories, Inc.

SEMI-VOLATILE LABORATORY
INSTRUMENT NUMBER 195 2004 WATER
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Reference Citation	Date Analyzed	Amount Spiked	Units	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Rep. #6	Rep. #7	Average Amount Found	Average % Recovery	Standard Deviation	MDL
N-Nitrosodimethylamine	8270C	12/29/2003	1.00	ug/L	0.550	0.490	0.370	0.180	0.710	0.620	0.310	0.461	46%	0.185	0.582
Phenol	8270C	12/31/2003	0.500	ug/L	0.273	0.264	0.270	0.340	0.325	0.343	0.281	0.299	60%	0.0350	0.110
Aniline	8270C	12/29/2003	1.00	ug/L	0.450	0.400	0.330	0.260	0.550	0.470	0.290	0.393	39%	0.105	0.330
Bis (2-chloroethyl)ether	8270C	12/31/2003	0.500	ug/L	0.397	0.466	0.483	0.537	0.501	0.429	0.514	0.475	95%	0.0490	0.154
2-Chlorophenol	8270C	12/31/2003	0.500	ug/L	0.425	0.372	0.411	0.476	0.460	0.482	0.431	0.437	87%	0.0391	0.123
1,3-Dichlorobenzene	8270C	12/31/2003	0.500	ug/L	0.411	0.377	0.393	0.465	0.456	0.457	0.424	0.426	85%	0.0344	0.108
1,4-Dichlorobenzene	8270C	12/29/2003	1.00	ug/L	1.01	0.910	0.780	0.580	1.06	0.880	0.620	0.834	83%	0.184	0.578
Benzaldehyde	8270C	12/29/2003	1.00	ug/L	0.880	0.790	0.900	0.710	0.980	1.02	0.770	0.864	86%	0.114	0.357
Benzyl Alcohol	8270C	12/29/2003	1.00	ug/L	0.490	0.420	0.320	0.280	0.690	0.540	0.330	0.439	44%	0.146	0.458
1,2-Dichlorobenzene	8270C	12/29/2003	1.00	ug/L	1.04	0.870	0.740	0.590	1.10	0.870	0.610	0.831	83%	0.198	0.621
2-Methylphenol	8270C	12/31/2003	0.500	ug/L	0.488	0.454	0.521	0.416	0.516	0.539	0.400	0.476	95%	0.0542	0.170
Bis-(2-chloroisopropyl)-ether	8270C	12/31/2003	0.500	ug/L	0.512	0.476	0.496	0.701	0.578	0.519	0.502	0.541	108%	0.0775	0.244
Acetophenone	8270C	12/29/2003	1.00	ug/L	0.810	0.720	0.710	0.700	0.860	0.840	0.670	0.759	76%	0.0760	0.239
4-Methylphenol	8270C	12/31/2003	0.500	ug/L	0.345	0.422	0.474	0.451	0.449	0.435	0.409	0.426	85%	0.0416	0.131
N-Nitrosodi-n-propylamine	8270C	12/29/2003	1.00	ug/L	1.16	0.780	0.640	0.570	1.12	0.860	0.600	0.819	82%	0.242	0.761
Hexachloroethane	8270C	12/29/2003	1.00	ug/L	0.920	0.830	0.650	0.480	0.940	0.760	0.550	0.733	73%	0.179	0.563
Nitrobenzene	8270C	12/31/2003	0.500	ug/L	0.450	0.528	0.449	0.609	0.542	0.491	0.466	0.505	101%	0.0586	0.184
Isophorone	8270C	12/31/2003	0.500	ug/L	0.532	0.481	0.563	0.619	0.641	0.547	0.601	0.569	114%	0.0553	0.174
2-Nitrophenol	8270C	12/31/2003	0.500	ug/L	0.526	0.604	0.636	0.554	0.654	0.596	0.647	0.602	120%	0.0483	0.152
2,4-Dimethylphenol	8270C	12/31/2003	0.500	ug/L	0.367	0.315	0.375	0.295	0.374	0.398	0.364	0.355	71%	0.0366	0.115
Benzoic Acid	8270C	2/11/2004	5.00	ug/L	3.36	3.78	3.23	1.52	3.48	3.39	3.43	3.17	63%	0.747	2.35
Bis(2-chloroethoxy)methane	8270C	12/31/2003	0.500	ug/L	0.469	0.399	0.513	0.550	0.477	0.510	0.484	0.486	97%	0.0472	0.148
2,4-Dichlorophenol	8270C	12/29/2003	1.00	ug/L	0.840	0.710	0.530	0.440	0.900	0.840	0.510	0.681	68%	0.187	0.587
1,2,4-Trichlorobenzene	8270C	12/29/2003	1.00	ug/L	0.970	0.820	0.650	0.500	1.00	0.800	0.560	0.757	76%	0.194	0.611

Approved By: AD 1-22-09
QA Officer

Approved By: JMK 1/23/09
Area Supervisor

Attachment 23.9 (Table 7)
Demonstration of Capability Study Example

TriMatrix Laboratories, Inc.

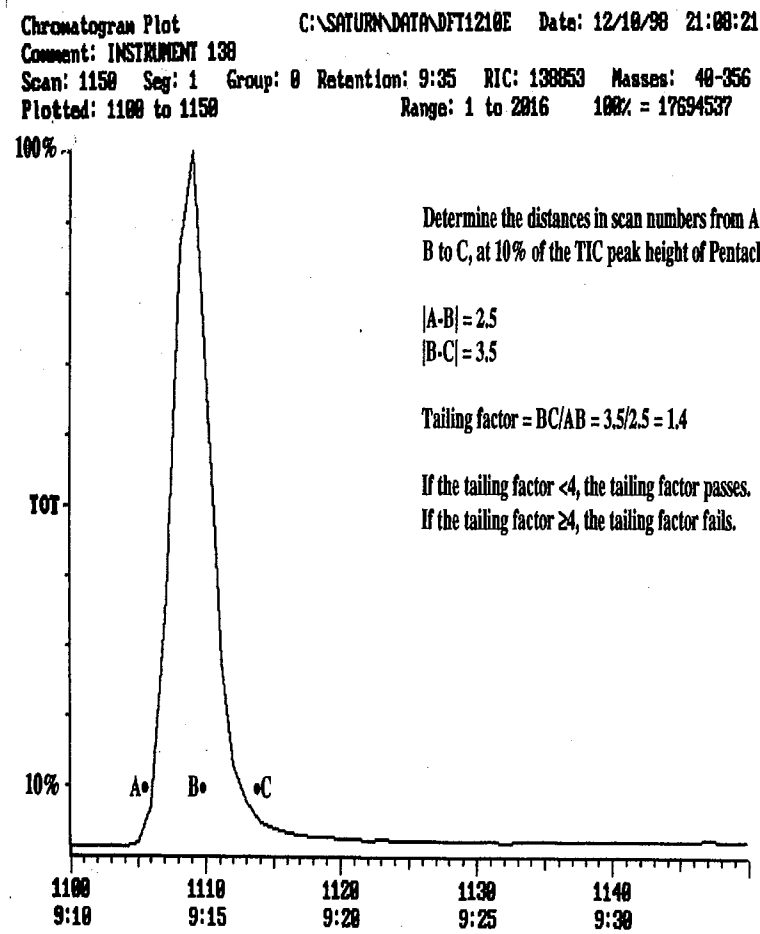
SEMI-VOLATILE LABORATORY
DEMONSTRATION OF CAPABILITY FOR SCOTT J. PUGH

Parameter	Date Analyzed	Method	Inst. Number	Units	Amount Spiked	Cert. #1 Amount Found	Cert. #2 Amount Found	Cert. #3 Amount Found	Cert. #4 Amount Found	Average Percent Recovery	Percent Recovery Window	Pass/Fail Percent Recovery	Percent RSD	Percent RSD Window	Percent RSD Pass/Fail	Overall Pass/ Fail
Phenol	12/15/2003	8270c	133	ug/L	10.0000	3.9900	3.9300	4.3400	4.0800	40.9%	19 -55	PASS	4.4%	20	PASS	*PASS*
Aniline	12/15/2003	8270c	133	ug/L	10.0000	5.2300	5.3400	5.8100	5.5100	54.7%	23 -84	PASS	4.6%	20	PASS	*PASS*
Bis(2-Chloroethyl)Ether	12/15/2003	8270c	133	ug/L	10.0000	8.9700	8.8900	9.6100	9.3900	92.2%	70 -130	PASS	3.7%	20	PASS	*PASS*
2-Chlorophenol	12/15/2003	8270c	133	ug/L	10.0000	7.8000	7.9400	8.8800	8.2500	82.2%	70 -130	PASS	5.8%	20	PASS	*PASS*
1,3-Dichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	7.4600	7.8600	8.2400	7.8000	78.4%	70 -130	PASS	4.1%	20	PASS	*PASS*
1,4-Dichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	7.1900	7.6200	8.2100	7.7500	76.9%	70 -130	PASS	5.5%	20	PASS	*PASS*
Benzyl Alcohol	12/15/2003	8270c	133	ug/L	10.0000	8.1300	8.2300	8.6500	8.7800	84.5%	70 -130	PASS	3.7%	20	PASS	*PASS*
1,2-Dichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	7.8900	8.0300	8.6100	8.2500	82.0%	70 -130	PASS	3.8%	20	PASS	*PASS*
2-Methylphenol	12/15/2003	8270c	133	ug/L	10.0000	7.5500	7.6700	8.0200	7.7800	77.6%	70 -130	PASS	2.6%	20	PASS	*PASS*
Bis(2-Chloroisopropyl)Ether	12/15/2003	8270c	133	ug/L	10.0000	9.3000	9.3700	9.7200	9.5600	94.9%	70 -130	PASS	2.0%	20	PASS	*PASS*
4-Methylphenol	12/15/2003	8270c	133	ug/L	10.0000	7.2900	7.1100	7.9500	7.7100	75.2%	70 -130	PASS	5.1%	20	PASS	*PASS*
N-Nitrosodi-N-Propylamine	12/15/2003	8270c	133	ug/L	10.0000	9.3500	9.2300	10.4000	10.0200	97.5%	70 -130	PASS	5.7%	20	PASS	*PASS*
Hexachloroethane	12/15/2003	8270c	133	ug/L	10.0000	7.8000	7.9000	8.3300	7.8200	79.6%	70 -130	PASS	3.1%	20	PASS	*PASS*
Nitrobenzene	12/15/2003	8270c	133	ug/L	10.0000	8.8400	9.3700	9.4300	8.9500	91.5%	70 -130	PASS	3.2%	20	PASS	*PASS*
Isophorone	12/15/2003	8270c	133	ug/L	10.0000	9.3100	9.7400	9.3600	9.5900	95.0%	70 -130	PASS	2.1%	20	PASS	*PASS*
2-Nitrophenol	12/15/2003	8270c	133	ug/L	10.0000	8.8100	9.5200	9.5200	9.3600	93.0%	70 -130	PASS	3.6%	20	PASS	*PASS*
2,4-Dimethylphenol	12/15/2003	8270c	133	ug/L	10.0000	7.2100	7.2400	7.0800	6.8600	71.0%	70 -130	PASS	2.4%	20	PASS	*PASS*
Benzoic Acid	12/15/2003	8270c	133	ug/L	10.0000	4.3400	5.4500	5.3600	5.5800	51.8%	20 -69	PASS	11.0%	20	PASS	*PASS*
Bis(2-Chloroethoxy)Methane	12/15/2003	8270c	133	ug/L	10.0000	9.1600	9.5800	9.5900	9.2700	94.0%	70 -130	PASS	2.3%	20	PASS	*PASS*
2,4-Dichlorophenol	12/15/2003	8270c	133	ug/L	10.0000	9.0800	9.3200	9.4000	9.3400	92.9%	70 -130	PASS	1.5%	20	PASS	*PASS*
1,2,4-Trichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	8.2000	8.6700	9.0400	8.5100	86.1%	70 -130	PASS	4.1%	20	PASS	*PASS*
Naphthalene	12/15/2003	8270c	133	ug/L	10.0000	9.2200	9.6100	9.3200	8.9200	92.7%	70 -130	PASS	3.1%	20	PASS	*PASS*

Approved By: _____ 1-22-09
QA Officer

Approved By: _____ 1/23/09
Area Supervisor

Attachment 23.10 (Figure 1)
Pentachlorophenol tailing factor



Determine the distances in scan numbers from A to B, and B to C, at 10% of the TIC peak height of Pentachlorophenol.

|A-B| = 2.5
|B-C| = 3.5

Tailing factor = BC/AB = 3.5/2.5 = 1.4

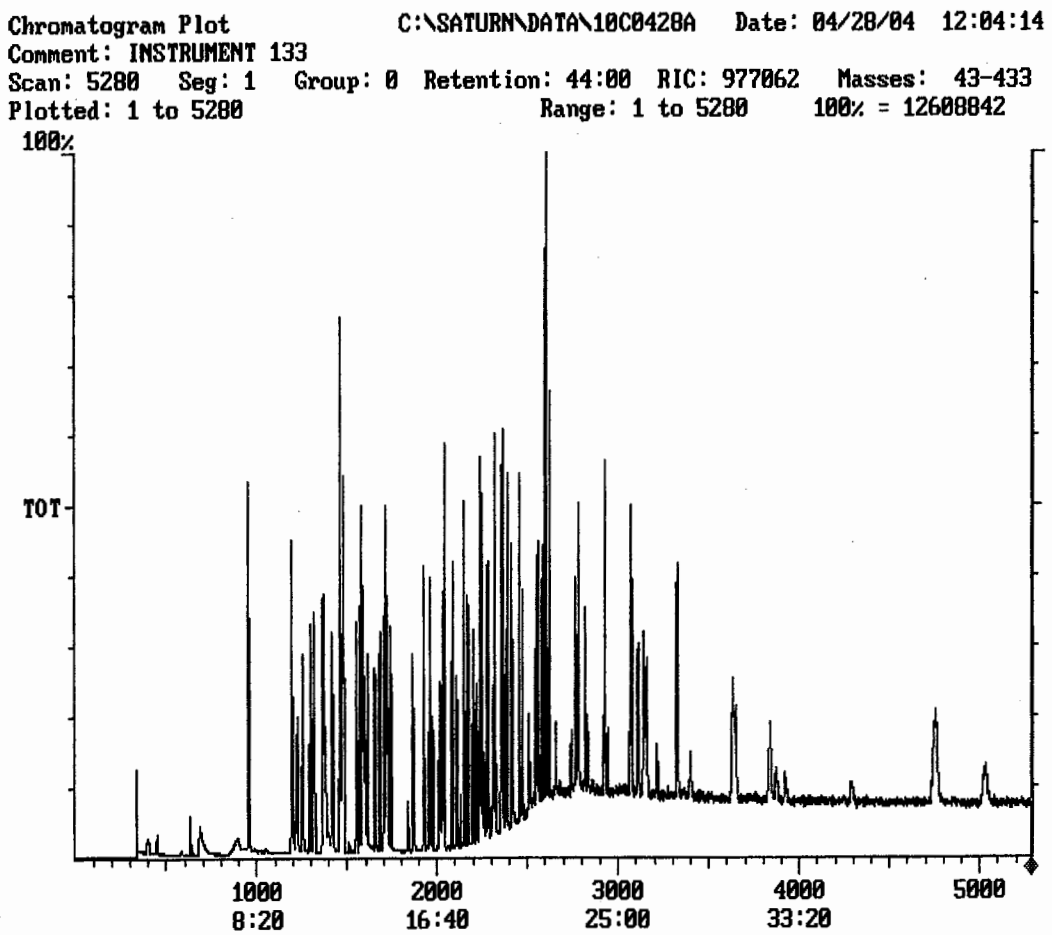
If the tailing factor <4, the tailing factor passes.
If the tailing factor ≥4, the tailing factor fails.

Approved By: _____ QA Manager

Approved By: _____ Area Manager

\\nt4vol1\project\sop\sv\current\gr04103.40

Attachment 23.11 (Figure 2)
Continuing Calibration Standard (10 ug/mL)
Total Ion Chromatogram, Varian Ion Trap

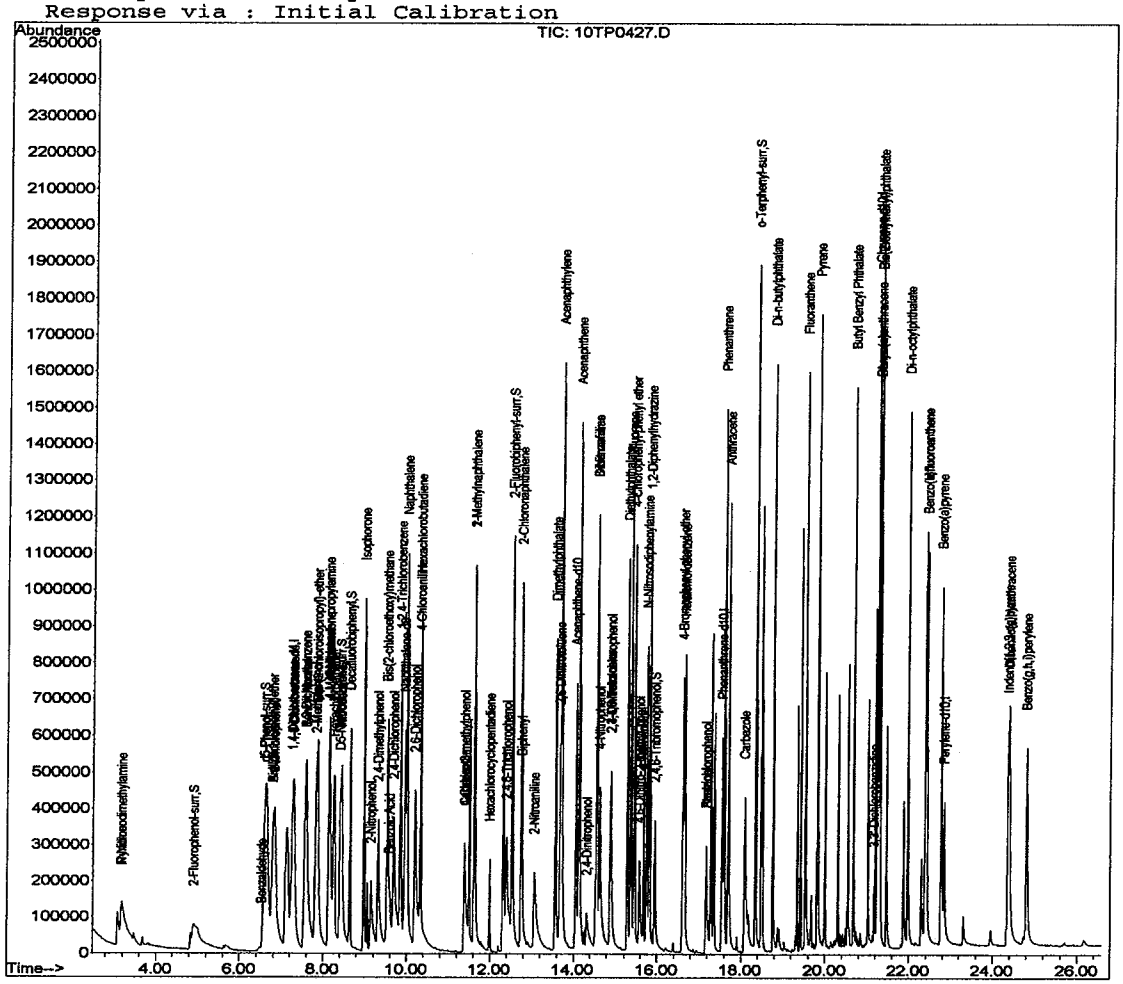


Attachment 23.12 (Figure 2A)
Continuing Calibration Standard (10 ug/mL)
Total Ion Chromatrogram, Agilent 5973 MSD

Quantitation Report

Data File : C:\HPCHEM\1\DATA\04-27-04\10TP0427.D Vial: 74
Acq On : 27 Apr 2004 10:17 Operator:
Sample : 10ppm bna pest Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Apr 27 10:44 2004 Quant Results File: BNA04074.RES

Method : C:\HPCHEM\1\METHODS\BNA04074.M (RTE Integrator)
Title : 8270C/625 BNA
Last Update : Tue Apr 27 17:24:56 2004
Response via : Initial Calibration



10TP0427.D BNA04074.M Thu Apr 29 14:34:16 2004 Page 4

Approved By: _____ 1-22-09 **Approved By:** _____ 1/23/9

QA Officer **Area Supervisor**

Attachment 23.13 (Figure 3)
Standards Form Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

A605395

Description:	DFTPP Tune Standard	Expires:	Aug-27-06
Standard Type:	MS Tune	Prepared:	May-11-06
Solvent:	Solvent Lot # C07H05	Prepared By:	Scott J. Pugh
Final Volume (mls):	1	Department:	Semivolatiles MS
Vials:	1	Last Edit:	May-11-06 09:04 by SJP

Solvent: MeCl

Analyte	CAS Number	Concentration	Units
4,4'-DDT	50-29-3	50	ppm
Benzidine	92-87-5	50	ppm
Decafluorotriphenylphosphine		5	ppm
Pentachlorophenol	87-86-5	50	ppm

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
A409098	Pentachlorophenol, SVMS	Aug-27-01	** Vendor **	Aug-27-06	Dec-01-05 11:57 by RGJ	0.05
A512731	4,4-DDT SVMS	Dec-08-05	** Vendor **	Dec-08-06	Dec-27-05 09:53 by SJP	0.05
A601262	Benzidine, SVMS	Feb-24-05	** Vendor **	Feb-24-08	Jan-09-06 15:23 by SJP	0.05
A603057	DFTPP, SVMS	Feb-27-06	** Vendor **	Feb-27-07	Mar-02-06 08:58 by SJP	0.005

Approved By: _____ 1-22-09 **Approved By:** _____ 1/23/09
QA Officer **Area Supervisor**

Attachment 23.14 (Figure 4)
Analytical Run Logbook Example

TriMatrix Laboratories, Inc.

Instrument ID: Saturn GC/MS Instrument #133 Date: 4/23/04 Taped: _____ Disk ID: _____ Analyst: SDS

Instrument Settings	Acquisition Information	Temperature Program					Miscellaneous
			Temp.	Rate	Time	Total	
Multiplier: 1550	Scan Range: 40-450 amu	GC	(C)	(C/min)	(min)	(min)	New Column Date: 4/6/4
AGC 7000	Seconds/Scan: 0.500						New Septa: Yes (No)
Interface Temp: 250C	Filament Delay: 3.5 min.	Step #1	45	0.0	1.5	1.5	Working Std. #: SV4.33-9
Source Temp: 190C	Threshold: 2 counts	Step #2	200	10	15.5	17.0	Int Std. #: SV4.31.5
Head Pressure: 8 psi	Mass Defect: 0	Step #3	315	25	4.6	21.6	Tune Std. #: SV4.23-8
	Background Mass: 35 amu	Step #4	315	0.0	16.4	38	Inj. Vol.: 1 uL
	Curve Date: 1/30/04	Inject.	45	0.0	0.5	0.5	DFTPP Area 198: 993078
		Inject.	300	100	2.55	3.5	DDT Breakdown: 0%
		Inject.	300	0.0	15	15.05	

Sample / File ID	Client	Inj Time	Dil	Matrix	Fraction	Method	Notes
DFT0423C		8:47		QC	DFTPP	8270	Passed
SC0423A		9:21		↓	BNA		Passed
BLK0423A		10:10	STR	QC			
358328	[redacted]	11:22		WTR			
358329		12:16					
358330		13:05					
358331		13:45					
358332		14:25					
358333		15:04					
358334		15:44					
358335	↓	16:24	↓	↓	↓	↓	
358336	[redacted]	17:04	STR	WTR	BNA	8270	

file: 133_RUN.XLS page: 16 of 61 revision: 1.0

Approved By: _____ 1-22-09 QA Officer Approved By: _____ 1/23/09 Area Supervisor

Attachment 23.15
Method 8270C Selected Ion Monitoring (SIM) Instructions

Selected Ion Monitoring (SIM) allows the mass spectrometer to detect specific compounds with very high sensitivity. In SIM mode the instrument obtains data at select masses of interest instead of stepping the mass filter over a wide mass range.

To set up SIM monitoring, the compounds of interest must first be analyzed by acquiring a full GCMS scan to determine the ions to use. Two to three ions are selected for each target compound. Include the most abundant ion if it is unique to the compound. Higher mass ions are usually more specific and separated from interference.

After choosing spectral fragments (ions) to monitor, an accurate mass must be determined (±0.1 AMU). The easiest way to determine mass accuracy is by tabulating the full-scan spectra of the compound. Print the mass table and use the m/z value in the tabulation to the nearest 0.1 AMU for the SIM acquisition mass.

After SIM acquisition masses are determined for each compound, ions are grouped according to elution time. Limit the number of ions being monitored in a group to between 3 and 8. Only one group can be monitored at a time. The end-time for one group becomes the start-time for the next group. Using these groups, the important ions for a list of compounds can be monitored by switching from one group to another at the appropriate time during a chromatographic run.

Once the monitoring ions are determined and the ions grouped, set a dwell time for each group to optimize the cycle time. Obtain 15 to 20 scans across each peak. A dwell time of 50 msec for each group is a good starting point. Acquire a run with this dwell time and note the number of scans across each peak. If the number of scans is less than 10, the dwell time needs reduced. If more than 25 scans, the dwell time needs increased.

Follow every step in the analytical procedure when using the SIM. A full-scan tune is acquired and must pass at the start of each 12-hour shift. Use a calibration curve of at least five points that pass acceptance criteria. Curve concentrations will depend on compound response and client requirements. Depending upon response, the low calibration standard is generally between 0.002 ug/mL and 0.1 ug/mL. Internal standard concentration is 0.5 ug/mL.

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Volatile Organic Compounds by Purge and Trap Capillary Column Gas Chromatography/Mass Spectrometry

EPA Method 624
SW-846 Method 8260B

APPROVALS:

Area Supervisor: Diane VanMale Date: 1-26-09
Diane L. VanMale

QA Officer: Date: 1-22-09
Tom C. Boocher

Operations Manager: Date: 1/26/09
Jeff P. Glaser

Procedure Number: GR-04-104
Revision Number: 4.5

Date Initiated: 9/1/95
Effective Date: 2/20/09

Date Revised: 1/22/09
Pages Revised: All

By: Diane L. VanMale
Total Number of Pages: 42

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure uses gas chromatography/mass spectrometry (GC/MS) for determining volatile organic compounds in a variety of matrices. It can be used to quantify most volatile organic compounds having a boiling point below 200° C. Volatile water-soluble compounds have higher quantitation limits due to poor purging efficiencies. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers and sulfides.

1.2 Nearly all matrices, including ground water, wastewater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments can be analyzed. Sample is introduced into the system using a purge and trap concentrator, or by direct injection if concentrations are extremely high.

1.3 For analysis of nonaqueous matrices, refer to TriMatrix SOP GR-04-105.

Note: Low-level bulk soil analysis by method 8260B is not allowed for state of Ohio samples under Ohio VAP (Voluntary Action Program) oversight and must not be used for Ohio VAP samples unless reported by method 8260A.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8260B, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 2, December 1996

2.2 *40 Code of Federal Regulations, most current edition*, Part 136, Appendix A, Method 624-Purgeables

2.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 8260A, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 1, September, 1994

2.4 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 5030B, "Purge and Trap for Aqueous Samples", Revision 2, December, 1996

3.0 SUMMARY OF PROCEDURE

3.1 When using purge and trap technique, an inert gas is purged through a sample, at an ambient or slightly elevated temperature. Volatile components are transferred from the aqueous to vapor phase. The vapor is swept onto a sorbent column, where volatiles are trapped.

3.2 After purging, the sorbent column is heated and back flushed with inert gas to desorb onto a capillary column. The capillary column is temperature programmed to separate and elute components, which are then transferred to a mass spectrometer, via jet separator or a direct connection, using an open-split interface or a capillary-direct interface with a split at the injection port.

3.3 Component analytes are detected by the mass spectrometer which is capable of qualitative and quantitative analysis. Identification is done by comparing analyte mass spectra against calibration standard spectra. Quantitation is performed by comparing analyte ion response (using an internal standard) to a minimum five-point calibration.

4.0 PARAMETER OR COMPOUND LIST

4.1 Compounds available for analysis and referenced to method 8260B or method 624 are listed in Attachment 23.1 (Table 1) and Attachment 23.2 (Table 1A).

4.2 Other analytes may be determined providing an acceptable demonstration of capability is performed following every step in the procedure. Additionally, all described quality control must be within laboratory established control limits.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-04-105, *Closed System Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples*, latest revision

5.2 TriMatrix SOP GR-03-124, *Volatile Laboratory Corrective Actions*, latest revision

5.3 TriMatrix SOP GR-15-102, *Waste Disposal,* latest revision

5.4 TriMatrix SOP GR-07-115, *Percent Solids, Gravimetric, Dried At 103-105° C*, latest revision

5.5 TriMatrix SOP GR-10-115, *Manual Integration*, latest revision

5.6 TriMatrix SOP GR-10-123, *Element™ Data Transfer and Review*, latest revision

5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Whether purged or injected directly onto a column, interferences naturally present in samples can elevate reporting limits and interfere with the analysis. Naturally occurring interferences can vary considerably from site to site.

6.2 Sample contamination can also raise reporting limits or give false positives. Contamination can come from a variety of sources. Improper sampling techniques can contaminate at the job site. During shipment and storage, volatile organics (particularly methylene chloride and fluorocarbons) can diffuse through septa. A trip blank prepared from reagent water and carried through sampling and handling serves as a check on such contamination. All volatile sample storage units must contain a storage blank, which must be replaced and analyzed weekly.

6.3 During analysis, contamination can come from impurities in the purge gas or from organic compounds outgassing from the plumbing ahead of the trap, if deposited by a previously analyzed high-level sample. To minimize, the non PTFE tubing, non PTFE thread sealants, and flow controllers with rubber components in the purging device, have been eliminated where possible.

6.4 Carryover can occur whenever high and low level samples are analyzed sequentially. To reduce carryover, the autosampler rinses the purging device and sample syringe with reagent water between samples. Samples with unusually high concentrations must be followed by analysis of a reagent water blank to check

for carryover. If compounds present in high concentrations are present at any level in a subsequent sample, demonstrate that there is no carryover by reanalyzing the sample. If compounds are not present in the subsequent sample, reanalysis is not necessary.

6.5 The trap and other parts are also subject to contamination. Frequent bakeout and purging of the entire system is required. If contamination persists, the complete purge and trap system must be purged first with 100° C reagent water then if necessary, with methanol. If methanol is used, disconnect the trap or install a blank trap, as methanol will adversely affect the trap's performance.

6.6 For samples containing large amounts of water-soluble material, suspended solids, high boiling compounds, or high organohalide levels, it may be necessary to wash out purging devices with a detergent solution, rinse with reagent water then dry in a 105° C oven between analyses.

6.7 Methanol content in blanks, standards, and samples must be kept constant on the ion trap. Varying methanol can suppress analyte signal response, and can alter certain spectra (chloroethane spectra will not look like the reference spectra).

Note: The maximum methanol volume purged must be no more than 100 µL.

6.8 Corrective actions for unacceptable quality control are outlined in TriMatrix SOP GR-03-124.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Safety Manual and Chemical Hygiene Plan.

7.2 The toxicity or carcinogenicity of reagents used has not been precisely defined. All chemicals must be treated as a potential health hazard. From this viewpoint, chemical exposure must be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel

7.3 The following analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromomethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds must be handled only in a hood, with disposable gloves, a laboratory coat and approved safety glasses.

7.4 Approved safety glasses must be worn in the laboratory except when inputting data at a computer terminal.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 To achieve reporting limits, a minimum of 40 mL aqueous sample (in duplicate) needs to be collected. More sample volume will be necessary if matrix spikes are required. Collect soil, sludge and solid waste samples as specified by TriMatrix SOP GR-04-105.

8.1.1 Collect all aqueous samples and liquid waste in 40 mL borosilicate glass screw-cap VOA vials with PTFE-lined silicone septa. All sample collection vials provided by TriMatrix contain acid preservative. Gently fill sample vials such that a meniscus develops at the top rim of the vial (fill to almost overflowing). To minimize analyte loss, no air bubbles must pass through a

sample as a vial is filled, or be trapped in the sample when the vial is sealed. Invert each filled and capped vial to confirm no headspace or bubbles are present. If there is headspace or bubbles larger than 5-6 mm in diameter, fill a new vial.

8.1.1.1 If vials were not supplied by TriMatrix and are not pre-preserved, fill to just overflowing, then adjust to a pH <2 by carefully adding two drops of 1:1 HCl to each vial. Seal the filled vials with a septa screw-cap lid (PTFE face must be down), then cool to 4 ±2° C.

8.1.1.2 If a sample contains residual chlorine, collect first in a 125 mL bottle, pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl the capped bottle to mix. Proceed with Section 8.1.1, then Sections 8.1.1.3 and 8.1.1.4 if applicable.

8.1.1.3 If analysis includes acrolein and/or acrylonitrile, and a project specifies it, collect additional sample in vials that do not contain preservative. Adjust to a pH of 4-5 with 1:1 HCl, then cool to 4 ±2° C.

8.1.1.4 Analysis on unpreserved or insufficiently preserved samples (where a sample has a high buffering capacity), which reference method 8260B, will not be conducted except by specific client or project request. In such cases, analysis will be conducted within 7 days, instead of 14.

Note: This is an uncommon exception to the 14-day holding time, handled on a case by case basis.

8.1.1.5 Method 624 samples may be collected with preservative as described above, or collected with no preservative. In either case, samples must be cooled to 4 ±2° C.

8.1.1.5.1 If collected without preservative, and acrolein is an analyte, sample analysis must be completed within 3 days. Acrylonitrile has a hold time of 14 days whether preserved or not. Regardless of preservation, acrolein and/or acrylonitrile results must be qualified as screening data only under method 624.

8.1.1.5.2 Samples collected for purgeable *aromatic* hydrocarbons analysis can be collected without a chemical preservative. However, the analysis must be completed within 7 days of sample collection.

8.1.1.5.3 Samples collected for purgeable *chlorinated* hydrocarbons analysis *only* can be collected without a chemical preservative. Analysis must be completed within 14 days of sample collection.

8.1.2 At least one trip blank must accompany each aqueous sample set collected from the same general site at approximately the same time, to the site and back. A trip blank is a pre-preserved 40 mL VOA vial filled at the laboratory with reagent water, sealed, and shipped to the sampling site with empty sample containers.

8.1.3 For trip blanks associated with soil, sludge and waste collection, refer to TriMatrix SOP GR-04-105

8.2 Aqueous samples and soil samples to be prepared following TriMatrix SOP GR-04-105 must be chilled to 4 ±2° C on the day of collection, and maintained at that temperature until received by the laboratory. Samples not received by the laboratory on the day of collection, must be packaged for shipment with sufficient ice to ensure a transit temperature of 4 ±2° C.

8.2.1 Once received, TriMatrix must store samples at 4 ±2° C until analysis. Aqueous, solid, and waste samples are stored separately. Each storage area must be free of organic solvent vapors.

8.2.2 With certain exceptions (noted in Sections 8.1.1.4 and 8.1.1.5), all samples must be analyzed within 14 days of collection or qualified as estimated.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Glassware and Hardware

9.1.1 Class A volumetric flasks (10 mL, 50 mL, 100 mL, 250 mL, 1000 mL)

9.1.2 Microsyringes (1.0 µL, 5.0 µL, 10 µL, 25 µL, 50 µL, 100 µL, 1000 µL)

9.1.3 Gastight luer lock syringes, 5.0 mL

9.1.4 60 or 125 mL wide mouth glass jars with PTFE-lined caps

9.1.5 Borosilicate glass vials with PTFE-lined septum caps, 20 mL and 40 mL

9.1.6 Various size PTFE-lined screw cap vials

9.1.7 Mini-inert vials, 1.0 mL for standards preparation and storage

9.1.8 Refrigerator, capable of maintaining 4 ±2° C

9.1.9 pH test strips

9.1.10 Metal spatulas

9.1.11 Analytical balance capable of reading to 0.0001 g

9.1.12 Top-loading balance capable of reading to 0.01 g

9.1.13 Pasteur pipettes (disposable) with rubber pipette bulb

9.2 Purge and trap/gas chromatograph/mass spectrometer system:

9.2.1 Concentrators

9.2.1.1 Encon (Environmental Sample Technology) concentrator conditions used:

Trap: EST Vocarb 3000
Purge: 11 minutes at 38-42 mL/minute
Dry purge: 1 minute

Desorb preheat: 245° C
Desorb: 250° C for 2.0 minutes
Bake: 10 minutes at 260° C
Valve oven: 150° C
Transfer line: 150° C

9.2.2 Autosamplers:

9.2.2.1 Archon (O.I. Analytical 4552 or Varian) – with needle sparger for heated purging of water and low-level soil samples; preheat for 1.0 minute and purge at 45° C

9.2.2.2 Centurion (Environmental Sample Technology) for water samples and MeOH preserved soil samples

9.2.3 Gas Chromatograph

9.2.3.1 Hewlett Packard Model 5890 Series II with a split/splitless injection port and a capillary direct interface. Conditions used:

Injector temperature: 200° C
Transfer line temperature: 280° C
Column temperature program:45° C for 10 minutes, then to 73° C at 7° C/minute, then to 110° C at 10°, then to 220° at 20° C/minute, hold 2.25 minutes .

9.2.3.2 Hewlett Packard Model 6890 equipped with electronic pressure control (EPC), a split/splitless injection port and a capillary direct interface. Conditions used:

Injector temperature: 200° C
Transfer line temperature: 280° C
EPC setting: Constant flow mode at 0.7 mL/minute
Split ratio: 50:1
Column temperature program: 45° C for 3 minutes, then to 200° C at 15° C/minute, hold for 1 minute

9.2.4 Columns:

9.2.4.1

9.2.4.2 30 m x 0.25 mm ID, 1.4 um film thickness, narrow bore capillary column DB-VRX (J&W Scientific). Used in HP 5890 II with capillary direct interface (Section 9.2.3.1).

9.2.4.3 20 m x 0.18 mm ID, 1.0 um film thickness, narrow bore capillary column DB-624 (J&W Scientific). Used in HP 6890 Series II with capillary direct interface (Section 9.2.3.3).

9.2.5 Mass Spectrometer (Hewlett Packard 5971A or 5973 MSD) capable of scanning from at least 35-650 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact mode, and producing a mass spectrum that meets all criteria in Attachment 23.3 (Table 2), BFB Key Ion Abundance Criteria, when 50 ng of 4-bromofluorobenzene (BFB) are purged or injected onto the analytical column.

GC/MS operating conditions:

9.2.5.1 Electron energy: 70 volts (nominal)

9.2.5.2 Mass range: 35-300 amu

9.2.5.3 Scan time: 1.5 scans/second

9.2.5.4 Source temperature: 160 - 220° C

9.2.6 Data Acquisition:

9.2.6.1 The HP MSD data acquisition system is a DOS-based HP Chemstation equipped with EnviroQuant environmental data analysis software. It is also capable of plotting EICPs and has a 120,000 compound NIST spectral library.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 GC Maintenance

10.1.1 Change septa weekly or as needed

10.1.2 Change gas scrubber traps yearly or as needed

10.1.3 Clip or replace GC column as needed

10.1.4 Check column head pressure daily

10.1.5 Check gas cylinder pressure daily and change if needed

10.1.6 Replace injection port liner as needed

10.2 Mass Spectrometer Maintenance

10.2.1 Mass Selective Detector (MSD) Maintenance Schedule

10.2.1.1 Drain and replace rough pump oil every 6 months

10.2.1.2 Check diffusion pump oil annually and change if discolored or low

10.2.1.3 Clean ion source at least annually (may be needed more frequently as shown by detector performance)

10.2.1.4 Examine and replace large o-ring seal on 5971 if worn

10.2.1.5 Replace electron multiplier as needed (maximum voltage 3000)

10.2.1.6 Check calibration gas and refill as necessary

10.3 Purge and Trap Maintenance Schedule

10.3.1 Before initial use, new traps must be conditioned for one hour.

10.3.2 Check and record the purge flow weekly

10.3.3 Empty waste bottles and fill rinse bottles daily

10.3.4 Check and record the concentrator pressures weekly

10.3.5 Check and if necessary fill internal/surrogate standard module daily

10.3.6 Clean sparge tube as necessary.

11.0 CHEMICALS AND REAGENTS

11.1 Reagent water (organic free) ASTM Type II

11.2 Methanol (purge and trap grade)

11.3 Certified stock standard materials (96% pure or greater) or certified stock solutions

11.4 1:1 Hydrochloric acid

11.5 Sodium bisulfate, $NaHSO_4$, ACS reagent grade or equivalent

12.0 STANDARDS PREPARATION

12.1 All laboratory standards must be recorded in the standards logbook and/or in the laboratory information management system (Element™). Information including the vendor, lot number, concentration, purity must be recorded. Each standard is given a unique ID number. All vendors and standard concentrations provided below are recommended and subject to change.

12.2 Hold Time and Storage Requirements

12.2.1 Unopened ampoules of purchased standard solutions and neat materials used in stock standards preparation, may be kept for up to 12 months from the date received or as directed by the manufacturer.

12.2.2 Due to evaporative loss and reactivity, laboratory prepared stock, and nongaseous intermediate stock standards are given an expiration date of six months. An intermediate stock gas standard typically requires weekly replacement. Within expiration dates, all standards must not be kept past acceptable performance.

12.2.2.1 Continuing calibration standards must meet the calculated percent difference or drift criteria of Section 14.6 to be acceptable. This requirement applies to all analytes, both gaseous and non-gaseous. Once this criterion has been exceeded, the intermediate stock and/or the stock must be replaced unless exceedance is due to instrument performance degradation.

12.2.2.2 Dichlorodifluoromethane will generally be the first gaseous compound to fail.

12.2.2.3 Because 2-chloroethyl vinyl ether is reactive, it will typically be among the first of the nongaseous compounds to fail.

12.2.3 Store all prepared standards with minimal headspace, protected from light, in PTFE sealed screw-cap vials at -10° C or below. Purchased standards are stored as instructed by the manufacturer. Standards must be stored separately from samples.

12.3 Stock Standard Preparation

12.3.1 Stock standards are prepared from neat materials or certified solutions. All purchased solutions must be accompanied by a Certificate of Analysis. Two separate standard sources must be obtained. This applies to both purchased standards and neat compounds. One set will be used to prepare calibration, laboratory fortified blanks (blank spikes) and matrix spikes, and the other will be used for laboratory control samples secondary calibration verifications). The two must be prepared from dissimilar or entirely separate sources. It is permissible to purchase different lots from the same vendor. However, a dissimilar lot means preparation from dissimilar chemical lots. Not prepared twice from the same lot. It is not necessary to purchase/prepare Internal Standard/Surrogate mixes from separate sources.

12.3.2 When preparing stock standards from neat material, the mass weighed out may be used without mathematical correction when the compound has a purity greater than or equal to 96%. Prepare stock standard solutions in methanol.

12.3.3 Gravimetric Method:

12.3.3.1 A 10,000 mg/L stock solution: 0.5 g of each solid analyte (neat) is transferred to a 50 mL volumetric flask partially filled with methanol which has been tared on an analytical balance. Record mass to the nearest 0.0001 g, make sure the solid is dissolved then fill to the mark with methanol. Cap and invert three times to ensure proper mixing. Discard the contents in the flask's neck and transfer the remaining solution to a PTFE-capped vial.

12.3.4 Purchased Commercial Stock Standards:

12.3.4.1 The following standards are combined to make Stock Standard I.

- Mix A: Volatile Organic Compounds - Liquids @ 2.0 mg/mL in MeOH
- Mix B: Volatile Organic Compounds - Gases @ 2.0 mg/mL in MeOH
- Mix C: Acrolein & Acrylonitrile @ 2.0 mg/mL in MeOH
- Mix D: Ketone Mix @ 2.0 mg/mL in MeOH
- Mix E: Custom Additions to Method 8260B @ 2.0 mg/mL in MeOH
- Mix F: Dichlorofluoromethane @ 1.0 mg/mL in MeOH

- Mix G: Internal Standard and Surrogate Standard Mix at 5.0 mg/mL in MeOH.
- Mix H: BFB Standard Mix at 2.0 mg/mL in MeOH.

12.3.4.2 The following standards are combined to make Stock Standard II.

- Mix A: Appendix IX Standard @ 2.5 mg/mL in MeOH
- Epichlorohydrin at 1.0 mg/mL in MeOH.
- 2-Chloro-1,3-butadiene (chloroprene) at 2.0 mg/mL in MeOH.
- N-butyl acetate at 2.0 mg/mL in MeOH.
- Methyl Methacrylate at 2.0 mg/mL in MeOH.

12.4 Intermediate Stock Standards

12.4.1 8260B Intermediate Stock Standard I

12.4.1.1 This solution is prepared in a 1.0 mL mini-inert vial. Add 650 µL of methanol to the mini-inert vial. From the mixes specified in Section 12.3.4.1, add the following volumes to produce 1.0 mL of a 100 mg/L intermediate stock standard:

50 µL Mixes A, B, C, D, and E
100 µL Mix F

12.4.2 Appendix IX Intermediate Stock Standard II

12.4.2.1 This standard is also prepared in a 1.0 mL mini-inert vial. Add 425 µL of methanol to the mini-inert vial. From the mixes specified in Section 12.3.4.2 and the neat stocks from 12.3.3.2, adding the following quantities will produce 1.0 mL of a 250 mg/L intermediate stock standard:

40 µL Mix A
100 µL Epichlorohydrin
50 µL 2-Chloro-1,3-butadiene
100 µL Isopropanol
100 µL Ethyl methacrylate
50 µL Methyl methacrylate
50 µL N-Butyl acetate
100 µL Cyclohexanone

12.5 Working Standards

12.5.1 Internal Standard/Surrogate Intermediate Stock Standard III

12.5.1.1 This standard is prepared at a concentration of 200 mg/L. Fill a 25 mL volumetric flask approximately three-quarters full with methanol. Add 1.0 mL of mix G specified in Section 12.3.4.1 and dilute to volume with methanol. Cap and invert three times to ensure proper mixing. Discard the contents in the flask's neck, and

transfer the remaining contents to a PTFE-capped vial. The purge and trap auto sampler adds this standard automatically to samples during the purge cycle.

12.5.2 BFB Intermediate Stock Standard IV

12.5.2.1 This standard is prepared in a 1.0 mL mini-inert vial. Add 990 µL of methanol to the mini-inert vial. Add 10 µL of mix H specified in Section 12.3.4.1 to produce 1.0 mL of a 20 mg/L intermediate stock standard. This standard is used for performing BFB tunes.

12.5.3 Initial calibration and calibration verification standards are prepared by spiking different volumes of intermediate stock standard into a 50 mL volumetric flask or a 5.0 mL gas-tight syringe, depending on the auto sampler used. These standards are prepared as needed and not stored. Calibration verification standards are typically prepared at 40 ug/L for all except Appendix IX compounds, which are prepared at 100 ug/L. Default spike volumes used in preparing the initial and continuing calibration verification standards are discussed in Section 14. Stock volumes necessary to prepare other concentrations can be calculated using the following formula:

$$V_s = \frac{C_f \times V_f}{C_s}$$

where:
V_s = Volume of stock standard to inject (µL)
C_f = Final concentration of working standard (ug/L)
V_f = Final volume of working standards (mL)
C_s = Concentration of stock standard (ug/mL)

12.6 Matrix Spike Standards

12.6.1 The second source intermediate stock standards prepared in Section 12.4 are used for matrix spiking. Matrix spikes are prepared using the formula given in Section 12.5.3. Matrix spike standards must be prepared at concentrations near the midpoint of the calibration, typically the same concentration as the continuing calibration standard. Other concentrations may be specified for certain projects.

12.6.2 The following analytes are default matrix spike compounds: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene and benzene. Additional analytes are reported if requested by clients.

12.6.2.1 Samples that must meet data quality objectives of the State of Wisconsin must be spiked with all target analytes.

12.6.2.2 Samples the must meet data quality objectives of the Department of Defence must be spiked with all target analytes.

12.6.2.3 Any client sample requiring all target analytes to be quality control spiked must be input to the laboratory information management system (Element™) to spike all target analytes.

12.7 Laboratory Fortified Blank/Blank Spike (LFB/BS) Standards

12.7.1 Primary source intermediate stock standards prepared in Section 12.4 are used for blank spikes. An LFB/BS is prepared using the calculation given in Section 12.5.3. LFB/BSs must be prepared at the same concentration as the matrix spike.

12.7.2 The same analytes used in matrix spikes must be used for the LFB/BS. Additional analytes are reported if requested by the client. The same analyte list must be reported for matrix spikes and the LFB/BS.

12.7.2.1 Samples that must meet data quality objectives of the State of Wisconsin must be spiked with all target analytes.

12.7.2.2 Samples the must meet data quality objectives of the Department of Defence must be spiked with all target analytes.

12.7.2.3 Any client sample requiring all target analytes to be quality control spiked must be input to the laboratory information management system (Element™) to spike all target analytes.

13.0 SAMPLE PREPARATION

13.1 Soils are prepared in accordance with TriMatrix SOP GR-04-105 with reference to SW-846 method 5035.

13.2 Waters are prepared using the Archon or Centurion Autosampler

13.2.1 Samples analyzed on these autosamplers require no preparation. The 40 mL vial is loaded directly into the autosampler.

13.2.2 If a dilution is required, the sample must be diluted into the appropriate size volumetric flask and an aliquot transferred to a 40 mL VOA vial for analysis. An ideal dilution results in an analyte concentration in the upper half of the calibration range.

13.2.3 The autosampler withdraws a five mL aliquot from the 40 mL vial, and adds the required internal standards and surrogates before transferring to the sparge vessel.

14.0 CALIBRATION PROCEDURES

14.1 Initial Calibration

14.1.1 A 7-point calibration must be run before sample analysis. The low point of the initial calibration curve must be at or below the minimum reporting limit for each analyte. The high point defines the linear range. Calibration standards must be matrix matched to samples whenever possible, and analyzed under the same operating conditions. All target compounds must be included in the initial calibration. A calibration must be analyzed only after a successful BFB analysis.

14.1.2 For the Archon or Centurion autosampler, prepare standards in 50 mL volumetric flasks using 10 times the volumes listed in Table 3 and 3A. Transfer to a 40 mL vial, then load in the autosampler. The autosampler takes a 5.0 mL aliquot and adds 1.0 µL of internal

standard/surrogate mixture before purging. The Archon and Centurion are used to analyze aqueous samples, and methanol extracts after dilution into reagent water.

Note: **SODIUM BISULFATE ADDITION DEGRADES 2-CHLOROETHYL VINYL ETHER IN STANDARDS AND SAMPLES. TRIMATRIX LABORATORIES HAS DEMONSTRATED THAT ACCEPTABLE RESULTS FOR THIS COMPOUND ARE NOT ACHIEVED WITH THIS TECHNIQUE.**

14.2 Calculate Response Factors (RF) for each compound using the quantitation and internal standard ions listed in Attachments 23.1 or 23.2. Calculate RF using the following formula:

$$RF = \frac{A_x \times C_{is}}{A_{is} \times C_x}$$

where:
A_x = Area of the characteristic ion for the target compound
A_{is} = Area of the characteristic ion for the specific internal standard
C_x = Concentration of the compound being measured
C_{is} = Concentration of the specific internal standard

14.2.1 For compounds not listed in Attachments 23.1 or 23.2, choose a major ion that is dissimilar to any potential coeluting or interfering ions, for the quantitation ion. Choose an internal standard with a retention time closest to the compound being measured.

14.2.2 Calculate average response factors as follows:

$$RF_{avg} = (RF_1 + RF_2 + RF_3 + \ldots..RF_n)/n$$

where:
RF_{avg} = Average calibration response factor
RF_1 = Calibration response factor for standard 1
RF_2 = Calibration response factor for standard 2
RF_3 = Calibration response factor for standard 3
RF_n = Calibration response factor for standard n
N = number of calibration standards

14.3 The average RF for all calibration points must be calculated for each compound. Five System Performance Check Compounds (SPCCs) must be checked against a minimum average RF limit.

14.3.1 These SPCCs are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. These minimum acceptable average response factors are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	>0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

14.3.2 An SPCC monitors compound instability and degradation caused by contaminated lines or active sites in the system. Examples of such occurrences are:

14.3.3 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

14.3.4 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

14.3.5 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated purge-and-trap transfer lines and/or active sites in trapping materials.

14.3.6 All non-SPCC compounds must adhere to the following minimum average RF limits:

14.3.6.1 The average RF of Alcohols, 1,4-Dioxane, and Epichlorohydrin must be at least 0.001 to be acceptable.

14.3.6.2 The average RF of all other compounds must be 0.010 or higher.

14.4 Always check for carryover and memory effects (ghosting) from high concentration standards or samples when analyzing for late eluting compounds such as hexachlorobutadiene and 1,2,3-trichlorobenzene. Adequate purge chamber rinsing minimizes such effects. Newer purge-and-trap systems address this problem with a bakeout step following trap desorb and newer traps retain less moisture. However, high concentrations can still affect subsequent runs.

14.5 Calculate Percent Relative Standard Deviation (%RSD) for all compounds. Using the initial calibration average RF, calculate percent RSD using the following formula:

$$\text{Percent RSD} = \frac{SD}{X} \times 100$$

where:

RSD = Relative standard deviation.
SD = Standard deviation of average RF for a compound.
X = Mean of the five initial response factors for a compound.

14.5.1 All compounds must have a percent RSD of ≤15%. Six compounds are used as Calibration Check Compounds (CCCs): 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethyl benzene, vinyl chloride. These compounds **must** have a percent RSD of ≤30%. If percent RSD is greater than 30 percent for any CCC, corrective action must be initiated and the entire system recalibrated.

14.5.1.1 If percent RSD of a compound is 15% or less, the calibration is assumed to be linear through the origin, and an average response factor may be used for quantitation.

14.5.1.2 If percent RSD of any compound is greater than 15%, then a regression curve of area ratio (Area analyte/Area IS) against concentration must be constructed, using first or higher order regression fit. Analysts must select a regression algorithm providing the best fit.

14.5.1.2.1 Generally this will be a first order linear regression using the following equation:

$C_i = A_i\text{-}b)/m$

Where:
C_i = Concentration in ug/mL
A_i = Area ratio ($A_{analyte}/A_{IS}$)
b = Intercept of the regression curve
m = Slope of the regression curve
i = Compound "i"

14.5.1.2.2 Second order regression must only be used for analytes with a definite quadratic response, as results produced by this algorithm may give erroneous results at concentrations near the reporting limit. A minimum of six calibration points are required to use second order calibration curves. The Enviroquant data processing software performs all regression functions, and uses coefficient of determination (r^2) to measure calibration validity. To be considered an acceptable calibration curve, r^2 must be 0.990 or higher.

14.5.1.3 If initial calibration criteria are not achieved using all calibration points run, the lowest or highest point on the curve may be dropped, provided enough points remain for the calibration model chosen.

14.5.1.3.1 A minimum of six calibration points are required to use second order regression curves. Five date points are needed for first order.

14.5.1.3.2 The lowest calibration point must be at the analyte reporting limit. The low point can not be dropped without elevating reporting limits.

14.5.1.3.3 Dropping the highest point shortens the calibration range, which could lead to a greater number of sample dilutions.

14.6 Initial calibration verification must be performed immediately after the initial calibration by running a second-source calibration verification (SCV) standard at 40 ug/L.

14.6.1 The SCV must be run after each initial calibration.

14.6.2 The SCV must include all targeted analytes.

14.6.3 Acceptance criteria for all analytes are ±25% of expected value unless otherwise specified in the laboratory information management system (Element™).

14.6.4 If any SCV analyte fails, locate and correct the problem (up to and including remaking the SCV solution) then repeat the SCV analysis successfully.

14.6.5 If the second SCV attempt fails, review the initial calibration solutions and remake or obtain a new calibration stock then repeat the SCV successfully.

14.6.5 Sample analysis may not begin until an acceptable initial calibration and SCV have been run successfully.

14.7 Every 12 hours a 40 ug/L continuing calibration verification (CCV) containing each compound quantified must be run after the BFB (Section 14.1). If a calibration has just been run, then the 40 ug/L standard from the calibration is used. Analysis of the CCV verifies instrument sensitivity and confirms acceptability of the initial calibration curve. A CCV is verified the same as an initial calibration, by checking SPCCs and CCCs. The CCV must be analyzed and quantified against a regression fit or average RF under conditions identical to the initial calibration.

14.7.1 System Performance Check Compounds (SPCC): This is the same check applied during initial calibration. SPCC compound response factors must be as listed in Section 14.3.1 (Section 14.2 for calculating response factors) In addition, non-SPCC compounds must meet the initial calibration criteria (Section 14.3.6) If minimum response factors are not met, the system must be evaluated and corrective action taken before sample analysis begins. Possible problems include standards degradation, injection port contamination and/or buildup in the first six inches of the analytical column and active sites in the column or chromatographic system.

14.7.2 Calibration Check Compounds (CCCs): After SPCCs are met, CCCs are used to check initial calibration validity. Calculate percent drift or percent difference using the following equations:

Percent Drift $= [(C_c - C_1)/C_1]*100$

where:
C_1 = CCC concentration
C_c = Measured concentration using selected average RF or regression.

Percent Difference $= [(RF_{ccc} - RF_{avg})/ RF_{avg}]*100$

where:
RF_{avg} = Average Response Factor from Initial Calibration
RF_{ccc} = Response Factor from Continuing Calibration

14.7.3 If CCC percent drift or difference does not exceed ±20%, the initial calibration continues to be valid. If acceptance is not met, then corrective action must be taken. An acceptable CCC **MUST** be run before sample analysis can begin. If CCC compounds are not target analytes, then all target analytes must be used to meet the ±20% control limits.

14.7.4 For non-CCC compounds in the CCV, the following criteria must be used.

14.7.4.1 All percent differences or drifts must not exceed ±25%, with the following exceptions:

14.7.4.2 Alcohols, ketones, 2-Methylnaphthalene, 2-Chloroethyl vinyl ether, 1,4-Dioxane, Vinyl acetate, and Iodomethane must not exceed ±40%.

14.7.4.3 Also, if non-CCC compound responses are high and out of control, then non-detect sample results for those compounds may still be reported since there is no question of analyte sensitivity for that day.

14.7.5 It is not permitted to choose a quantitation technique dissimilar to that used during initial calibration to achieve CCC criteria. For example: An analyte initial calibration %RSD was within ±15% drift, and average RF is used for quantitation. The last CCC is not acceptable for percent drift. To attain a passing CCC, it is not permitted to switch to a regression curve in an attempt to pass the CCC, even if the regression curve passes coefficient of determination criteria.

14.7.6 Internal standard responses and CCV retention times must be evaluated during or immediately after data acquisition.

14.7.6.1 If retention time for an internal standard changes by more than 30 seconds compared to the 40 ug/L initial calibration, the system must be inspected for malfunctions. Corrections must be made as needed.

14.7.6.2 If the area for an internal standard changes by a factor of two (-50% to +100%) compared to the 40 ug/L initial calibration standard, the system must be inspected for malfunctions. Corrections must be made as needed.

14.7.6.3 When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

15.0 ANALYTICAL PROCEDURE

15.1 Before initial use, a Vocarb 3000 trap must be conditioned for at least one hour by baking at 260° C and purging with helium. If other trapping materials are substituted for the Vocarb 3000, follow the manufacturer's conditioning recommendations. After periods of inactivity, GC columns may be run through the temperature program, or ramped to the final temperature and held for 15 minutes.

15.2 Set up the autosampler, purge-and-trap, and GC/MS as instructed in Section 9.0. Sample prep and purging conditions are specific to each matrix. The procedure for soil samples is described in TriMatrix SOP GR-04-105.

15.3 BFB Tuning

15.3.1 At the beginning of every 12-hour shift 4-Bromofluorobenzene (BFB) must be used to tune the mass spectrometer. Analyze the BFB and compare the spectra obtained to the criteria given in Attachment 23.3. No other analysis may begin prior to running an acceptable BFB tune.

15.3.2 The standard prepared in Section 12.5.2.1 contains 20 mg/L of 4-Bromofluorobenzene (BFB).

15.3.2.1 For the Archon autosampler, spike 25 µL into a 50 mL volumetric flask containing reagent water. Transfer the contents to a 40 mL sample vial. Alternately, 2.5 µL may be directly injected into the gas chromatograph.

15.3.2.2 The result will be to inject 50 ng of BFB into the GC/MS system. All subsequent analysis must commence within 12 hours of the BFB injection. BFB must be analyzed using the same acquisition parameters as standards and samples.

15.3.3 The following evaluation sequence must be used to determine BFB tune performance.

15.3.3.1 All BFB tunes must be initially evaluated using the CLP BFB tuning procedure, which takes the scan average at the peak apex and two scans immediately before and after the apex, followed by a background subtraction.

15.3.3.2 If the above does not generate an acceptable BFB tune, then take a scan average of the entire peak, followed by a background subtraction.

15.3.3.3 If the above two options do not yield an acceptable BFB tune, then evaluate with a single scan at the apex.

15.3.3.4 If none of the above give an acceptable BFB tune, then corrective action is required. The first step is to reinject and reanalyze the same BFB standard. Next, prepare a new BFB working solution, then inject and analyze. The last step is to optimize the tuning acquisition parameters by tweaking manually or by performing a new autotune. Once acquisition parameters are optimized, restart the tuning sequence.

15.4 Initial/Continuing Calibration

15.4.1 After a successful BFB analysis, an initial curve or continuing calibration verification must be analyzed (Section 14.0). Initial and continuing calibrations must match sample methanol and/or sodium bisulfate concentrations wherever possible, and be run under the same analytical conditions as samples. Follow initial or continuing calibration with the quality control described in Section 18.0.

15.5 Sample Analysis

15.5.1 For aqueous samples analyzed using the Archon or Centurion autosampler, the 40 mL sample collection vial is placed directly into the autosampler without opening. The autosampler removes 5.0 mL and transfers it to the purge vessel automatically. Internal standards and surrogate are added by the autosampler as well. The purge sequence will start automatically, using the operating conditions in Section 9.0. At the end of the purge cycle, analytes remain on the adsorbent trap, and are ready for desorption to the gas chromatograph.

15.5.2 Low concentration analysis for bulk and sodium bisulfate preserved soils (approximate concentration range 0.010 - 1.0 mg/kg):

15.5.2.1 If a sample is preserved in sodium bisulfate, remove the vial from storage, and let warm to room temperature. Agitate the vial gently so contents move freely and stirring will be effective. For bulk soils prepared in the laboratory, 5.0 mL of reagent water and a PTFE stir bar must be added during preparation. Place the vials in the autosampler.

15.5.2.2 The autosampler adds 10 mL of organic-free reagent water, internal standards, and surrogates before initiating the purge sequence. Other volumes may be used but **it is imperative that all samples, blanks and calibration standards have exactly the same added volume of organic-free reagent water**.

15.5.2.3 Before purging, vials are heated to 45° C and held for 1.0 minute. Purge follows the operating conditions provided in Section 9.0. Heat and stir using the magnetic

stir bar, for the entire purge cycle. After purging, analytes remain on the adsorbent trap, and are ready for desorption to the gas chromatograph.

15.5.2.4 This technique must be used only by client request.

15.5.4 The following is for extraction of high concentrations (greater than 1 mg/kg), from solid and oily waste:

15.5.4.1 Remove sample methanol extracts from storage and let warm to room temperature. Using a microsyringe, transfer an appropriate extract volume to organic-free reagent water in a 5.0 mL syringe or into a 50 mL volumetric flask. Minimally a 1:50 dilution is done.

15.5.4.2 Transfer the diluted extract to a 40 mL vial and place in the autosampler. The autosampler will add 10 mL of organic-free reagent water if using the Archon autosampler and internal standards/surrogates before purging.

15.5.4.3 Report soil results on a dry weight basis. Report waste results on a wet weight basis.

15.5 Sample Desorption

15.5.1 After the 11 minute purge, the system will automatically advance to the desorb mode. The trap will preheat to 245° C without desorption gas flow. When the trap reaches this setpoint, it will desorb at 20 mL/minute for two minutes. Desorbing initiates the gas chromatograph oven temperature program, and data acquisition begins.

15.6 After desorbing, the trap is reconditioned by baking out at 260° C (or the temperature recommended by the trap packing material manufacturer). After 10 minutes, the trap heater is turned off and purge flow through the trap is halted. When the trap is cool, the next analysis can begin.

15.7 If any compound response exceeds the calibration range, prepare a sample dilution. An ideal dilution will result in analyte response at midrange in the calibration. However, a dilution with a response in the upper 60% of the range is acceptable.

15.7.1 For aqueous sample dilutions, prepare a sample dilution from an aliquot of the second (duplicate) 40 mL vial or from unused sample saved under zero headspace from the first vial. A dilution may be prepared in a 5 mL syringe (for 5.0 mL purge volumes) or in a volumetric flask (50 mL or larger). The minimum volume that can be diluted is 1.0 mL. The total volume purged must equal the volume used for calibration standards (5.0, 10 or 25 mL). Organic-free reagent water must be used for all dilutions. Matrix-matching must be done by including the same methanol volume to dilutions as used in calibration standards. Pour the contents into a 40 mL vial then load into the autosampler, which will automatically add internal standards/surrogates.

15.7.2 For bulk soil dilutions, prepare a 1.0 g sample instead of 5.0 g if the expected concentration is within the calibration range. If a larger dilution is needed, refer to TriMatrix SOP GR-04-105.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Qualitative Analysis:

16.1.1 An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard. Standard spectra are obtained from calibration standards.

16.1.1.1 Two criteria must be satisfied to verify analyte identification:

16.1.1.1.1 Elution at the same GC relative retention time (RRT) as the standard.

16.1.1.1.2 Positive correlation of the sample analyte mass spectrum with the standard.

16.1.1.2 The sample component RRT must compare within ±0.06 RRT units of the daily continuing calibration standard. If coelution interferes with accurate component RRT assessment from the total ion chromatogram, the RRT must be assigned by using extracted ion current profiles, for ions unique to the compound.

16.1.1.3 All ions present in standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%), are automatically checked by the software to be present in the sample spectrum. Relative ion intensities must agree within plus or minus 20%, between standard and sample spectra.

Example: For an ion having a relative intensity of 50 percent in standard spectra, corresponding sample ions must be between 30 and 70 percent.

16.1.2 For sample compounds not associated with a calibration standard, a library search can be made for tentative identification. Only after visual comparison of sample spectra with the nearest library searches will an analyst assign a tentative identification. When a tentatively identified compound (TIC) cannot be identified by name, a generic description will be given to help identify functional groups. These TICs will have names such as:

Name	CAS #
Unknown Alcohol	xx-xx-xx
Unknown Freon	xx-xxx-xxxx
Unknown Ketone	xx-xxx-xx
Unknown Acid	xxx-xx-xxxx
Unknown Hydrocarbon	xxx-xxx
Unknown Glycol Ethers	xxx-xx-x
Unknown Substituted Benzenes	Xxxxx
Hydrocarbons (sub. Benzenes)	xxxxx-x-x
Hydrocarbons, Total	Xxxxxx

Guidelines for making a tentative identification are:

16.1.2.1 Relative intensities of major ions in the library spectrum (ions >10% of the most abundant ion) must be present in a sample spectrum.

16.1.2.2 Relative intensities of major ions must agree within ±20%. As an example: For an ion having a relative intensity of 50 percent in library spectra, corresponding sample ions must be between 30 and 70 percent.

16.1.2.3 Library spectra molecular ions must be present in a sample spectrum.

16.1.2.4 Sample spectra ions not present in the library spectrum must be reviewed for the presence of background contamination or coeluting compounds.

16.1.2.5 Library spectra ions not in sample spectra must be reviewed for possible subtraction from the sample spectrum, because of background contamination or coeluting peaks. Data system library reduction can sometimes create such discrepancies. When TIC searches are performed, the criteria for determining whether or not a peak is a TIC are:

16.1.2.5.1 The peak in question must be >10% of the nearest internal standard.

16.1.2.5.2 The top 10 potential TICs must be reviewed, unless otherwise required. The match quality must be 70 percent (corresponding to a fit of 700 on the Ion Trap) or higher, to report a positive identification in the absence of interfering peaks. If there are interfering peaks, analyst discretion must be employed when reporting positive matches.

16.1.2.5.3 Concentrations <1 ug/L or <0.01 mg/kg (assuming a nominal sample extraction volume or mass, and instrument response) will not be reported as a TIC unless otherwise specified for the project, and deemed achievable following supervisor data review.

16.1.2.6 Concentrations obtained must be reported indicating the values are estimated, and are flagged with an E. Calibrated analytes not part of a client's target analyte list will also be reported when TIC's are requested. They are to be given a Q flag (quantitated) if the concentration is within the calibration range or CE flag (curve estimated) if outside the range. If no valid identification can be made, the compound must be reported as an unknown aromatic, hydrocarbon or other class, if classification is possible.

16.2 Quantitative Analysis

16.2.1 Quantitation is performed using the same technique used during initial calibration (average response factor or regression). Use analyte area and internal standard ions as specified in Attachment 23.1 (Table 1A) and Attachment 23.2 (Table 1A), and the equations below.

16.2.1.1 Aqueous Samples

$$\frac{A_x \times C_{is}}{A_{is} \times RF} \times DF = ug/L$$

where:
A_x = Area of the characteristic ion for the target compound
A_{is} = Area of the characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard in ug/L
RF = Average Response Factor
DF = Dilution factor

16.2.1.2 Low Level Soil Samples

$$\frac{mg}{kg} = \frac{A_x \times C_{is}}{A_{is} \times RF} \times \frac{5\ mL}{W \times \%S} \times \frac{L}{1000\ mL} \times \frac{mg}{1000\ ug} \times \frac{1000\ g}{kg}$$

where additionally:
W = Wet weight of sample (g)
%S = Percent solids in decimal form (example: 0.90, not 90%). Used to calculate dry weight results for soils and sludges. Wastes are calculated on a wet weight basis only. Refer to TriMatrix SOP GR-07-115.

16.2.1.3 High Level Soil Samples

$$\frac{mg}{kg} = \frac{A_x \times C_{is}}{A_{is} \times RF} \times \frac{DF}{\%S} \times \frac{V}{W} \times \frac{L}{1000\ mL} \times \frac{mg}{1000\ ug} \times \frac{1000\ g}{kg}$$

where additionally:
V = Volume of solvent added to sample during purging or dilution (mL)
W = Wet weight of sample purged or diluted (g)

16.2.1.4 Equations given above will also be used to quantitate TICs, substituting total ion areas for A_x and A_{is}. The internal standard chosen must be the one closest in elution time to an unknown peak, provided it is free from most interferences. C_{is} remains the same and RF will be a default of 1.0.

16.3 All manual integration must be performed with strict adherence to TriMatrix SOP GR-10-115.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running sample sets are responsible for correctly filling in, handing in and filing associated paperwork. It is essential to perform these tasks to provide defensible data and client reporting.

17.2 Reporting to the laboratory information system must be done in accordance with TriMatrix SOP GR-10-123.

17.3 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in and archived correctly. The time each analyst has sample possession must be accounted for on the form.

17.3 All laboratory hardcopy (including CoC forms) must be archived appropriately and correctly.

17.3.1 All run and maintenance logbooks must be filled in completely and correctly. Corrections must be made with a lineout, not a writeover and must be dated and initialed. Blank lines must be Z'd out, initialed and dated.

17.3.2 All tunes, calibrations and continuing calibration verification runs must be placed in the correct archival folder.

17.3.3 All LIMS reports must be kept and placed in the correct archival folder.

17.4 The following are required for data packages requiring CLP-like deliverables:

17.4.1 Copies of all tunes including the EICP that has BFB ion peaks and ion abundance results and requirements, including mass listings, and associated Form Vs

17.4.2 Copies of all associated curves, including raw data quantitation reports, chromatograms, and associated Form VIs

17.4.3 Copies of all associated continuing calibration verifications, including raw data quantitation reports, chromatograms, and associated Form VIIs

17.4.4 Copies of all sample raw data, including all applicable quality control samples, quantitation reports, including triple plots for positive sample results, chromatograms and all associated TIC reports when requested

17.4.5 Method Blank Summary Form IVs

17.4.6 Internal standard recovery Form VIIIs

17.4.7 Copies of all applicable supporting information including: internal chain of custody forms, run and standard logbooks, method detection limits and extraction summaries

Note: For most projects, Forms I, II and III will be included with the deliverables package.

17.5 Rounding and significant figure adjustment is only performed on the final reported result by Element™.

18.0 QUALITY ASSURANCE

18.1 Quality control requirements include the following previously covered requirements and the quality control that follows:

18.1.1 BFB (Section 15.3)

18.1.2 Initial Calibration (Section 14.1)

18.1.3 Initial Calibration Vericiation (Section 14.6)

18.1.3 Continuing Calibration (Section 14.7)

18.2 Method Preparation Blanks (BLK)

18.2.1 After the BFB and initial calibration or continuing calibration have been run, a purged blank is required before sample analysis showing the analytical system to be free of interference and contamination.

18.2.2 The blank concentration must be at or below the maximum acceptance limit listed in the laboratory information management system. However methylene chloride and acetone may be up to five times the reporting limit unless further restricted by specific data quality objectives.

18.2.3 At a minimum, a BLK is run every 12-hour shift. The BLK must be run more frequently if carryover is suspected from a high concentration sample analysis or if laboratory contamination is in question. The BLK must be carried through all steps of sample preparation and analysis, including the addition of internal standards/surrogates spiking.

18.2.4 The BLK must be prepared in the same matrix as samples:

18.2.4.1 For methanol extractions, the BLK must be prepared as for a methanol extraction using a 1:50 ratio of methanol.

18.2.4.2 If samples are prepared with sodium bisulfate preservative, the BLK must also be prepared with sodium bisulfate.

18.2.4.3 For low-level soil analyses, the BLK must be prepared in a clean solid matrix.

18.3 Internal Standards

18.3.1 Internal standard responses and retention times in all runs following continuing calibration verification must be evaluated during or immediately after data acquisition. The retention time for internal standards must be within ±30 seconds from the current 12-hour continuing calibration standard. The quantitation ion area for all internal standards must stay within a factor of two (-50% to +100%) from the current 12-hour continuing calibration verification.

18.3.2 If at any time an internal standard fails the -50% to +100% area criteria, the ability to accurately quantitate analyte is reduced. Every effort must be made to prevent an internal standard failure, including sample dilution and reanalysis. Refer to TriMatrix SOP GR-03-124, for when an internal standard fails. If many samples are out-of-control for no apparent reason, the mass spectrometer must be inspected for malfunctions and appropriate maintenance performed. After maintenance has resolved the problem, samples run while the instrument was malfunctioning must be reanalyzed.

18.4 Surrogates

18.4.1 All samples and quality control must be spiked with surrogates. Until thirty samples of a given matrix have been analyzed, default recovery limits of 70 - 130% will be used. Once thirty samples of a given matrix have been analyzed, in-house recovery limits will be generated. At a minimum, surrogate acceptance limits must be updated annually on a matrix by matrix basis. Calculate surrogate recovery for each analysis. If recovery or precision is not within acceptable limits, consult TriMatrix SOP GR-03-124 to determine when and how data is qualified.

18.4.1.1 High recovery may be due to a co-eluting matrix interference from the sample. Examine the chromatogram for evidence of co-elution. No corrective action is required in this instance.

18.4.1.2 Low recovery may be due to poor purge efficiency. This should be verified by re-purging the sample if hold times permit.

18.4.1.3 If surrogate recovery in a purge blank (BLK) is below the lower control limit, only samples with failing surrogate recoveries will require re-purging. If BLK

surrogate recovery is above the upper control limit, no corrective action is required as long as sample surrogate recoveries are acceptable.

18.4.1.4 If surrogate recovery fails in the MS/MSD, re-analysis is only required if LFB/BS spike recovery also fails. If the LFB/BS is still out-of-control after re-analysis, all associated samples must be re-analyzed.

18.4.2 Calculate upper and lower control limits for each surrogate. This must be done as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

where
p = average percent recovery
s = standard deviation of the average

18.4.3 Two standard deviations will be used when three give a negative lower control limit.

18.4.4 If recovery fails, refer to TriMatrix SOP GR-03-124. If many samples are out-of-control for no apparent reason, the mass spectrometer must be inspected for malfunctions and appropriate maintenance performed. After maintenance has resolved the problem, samples run while the instrument was malfunctioning must be reanalyzed.

18.5 Laboratory Fortified Blank/Blank Spike (LFB/BS)

18.5.1 An LFB/BS is required with each 12-hour shift or with each batch of up to 20 samples, whichever is more frequent. The LFB/BS serves as a check of methanol extraction/purging efficiency should matrix spike recoveries not be within quality control limits.

18.5.1.1 When possible, the daily CCV doubles as the blank spike.

18.5.1.2 The LFB/BS is run at the beginning of the 12-hour shift following analysis of the BLK.

18.5.1.3 Recoveries must be calculated and compared to LIMS control limits. Analysis must be stopped and the problem corrected if recoveries are outside quality control limits. Samples analyzed in a batch with a failing LFB/BS must be reanalyzed for failed analytes. If this is not possible due to holding time or lack of sample, data for the failed analytes must be qualified as estimated.

18.5.1.4 An LFB/BS may be from the same source as the calibration or from the second-source used to verify the intial calibration.

18.5.2 At a minimum, the LFB/BS must contain the following analytes:

18.5.2.1 1,1-Dichloroethylene

18.5.2.2 Trichloroethylene

18.5.2.3 Chlorobenzene

18.5.2.4 Benzene

18.5.2.5 Toluene

Note: As noted previously, where project specifications require, all target analytes must be in the matrix spike, matrix spike duplicate and LFB/BS.

18.5.3 Until at least twenty blank spikes have been analyzed in a given matrix, recovery will be validated against default recovery limits of 70 - 130%. The blank spike must be prepared in the same matrix as samples:

18.5.3.1 For methanol extractions, the blank spike must be prepared as for a methanol extraction using a ratio of 1:50 of methanol.

18.5.3.2 If samples are prepared with sodium bisulfate preservative, the blank spike must also be prepared with sodium bisulfate.

18.5.3.3 For low-level soil analyses, the blank spike must be prepared in a clean solid matrix.

18.5.4 If an LFB/BS is out-of-control, the problem must be immediately identified and corrected before samples can be run.

18.5.4.1 The LFB/BS verifies that out-of-control compounds in an MS or MSD are the result of matrix interference rather than extraction or purging error.

18.5.4.2 Failure of any targeted analyte in an LFB/BS requires corrective action depending upon the failed parameter. Every effort must be made to determine the reason for failure (spike, extraction or purge error). Appropriate corrective action must then be taken.

18.5.4.3 Take corrective action in accordance with TriMatrix SOP GR-03-124.

18.5.4.4 Samples may not be reported without an acceptable LFB/BS.

18.6 Matrix Spikes (MS, MSD)

18.6.1 To assess extraction and purge efficiency from specific matrices, extract a Matrix Spike (MS/SPK) and Matrix Spike Duplicate (MSD/SPK) at least once every 20 samples prepared for each matrix. If less than 20 samples are analyzed in a month, at least one matrix spike and one matrix spike duplicate is required. Generally, matrix spikes are analyzed at the end of the 12-hour shift. Running matrix spikes at the end of the shift helps document the instrument is still functioning correctly.

18.6.2 Until at least twenty matrix spike/matrix spike duplicates have been analyzed, recovery will be validated against default recovery limits of 70 - 130%. The maximum default precision acceptance limit is 20% relative percent difference (RPD).

18.6.3 Once twenty MS/MSD sets of a given matrix (water, soil or waste) have been analyzed, statistical acceptance limits will be calculated by the laboratory information management system and listed there.

18.6.4 If MS/MSD recovery or duplication is not within acceptable limits, take corrective action in accordance with TriMatrix SOP GR-03-124.

18.7 This procedure is written primarily in reference to SW-846 8260B, however the following modifications are allowed when used for EPA method 624 sample analyses:

18.7.1 The 12-hour shift is replaced by a 24-hour shift. BFB and continuing calibration verifications are still required but only run once in 24 hours instead of every 12.

18.7.2 A 3-point calibration can be used in place of the 7-point.

18.7.3 SPCCs and CCCs are not used. Instead response factors for every compound listed on the EPA 624 list must have ≤35% RSD for the calibration to be valid.

18.7.4 The continuing calibration verification does not use SPCCs and CCCs. The RF for every compound in the 40 ug/L continuing calibration verification is compared with the corresponding calibration acceptance criteria found in Attachment 23.8 (Table 5). If parameter responses fall within the designated ranges, analysis of samples can begin. If any individual RF falls outside the range, a new continuing calibration verification or a new calibration must be run.

18.7.5 There are no criteria for internal standard areas or retention times in method 624. However, method 8260B criteria will be followed.

18.7.6 Every targeted analyte must be spiked in the MS/MSD, SCV and LFB/BS. Not just the limited list in Section 18.5.2.

18.7.7 Acrolein and Acrylonitrile may only be screened by GC/MS. All positive results must be qualified in the report as estimated.

18.7.8 Refer to Section 8.1.1.5 for sample collection requirements unique to method 624.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis to demonstrate acceptable accuracy and precision.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Spike four aliquots of organic-free reagent water so analyte concentrations are in the lower half of the calibration range. Process the four spikes following every step outlined in the procedure, including quality control.

19.1.1.2 Input all four results to the IDC spreadsheet located on the laboratory intranet library. Average percent recovery must be within the LFB/BS acceptability limits listed in the laboratory information management system. Relative standard deviation must be ≤20%.

19.1.1.3 If any criterion in the study fails, locate and correct the source of the problem and repeat the study successfully.

19.1.1.4 Repeated failure, will confirm a general problem with the procedure and/or techniques used. If this occurs, locate the problem and correct the procedure and/or techniques used then repeat the study successfully.

19.1.1.5 Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed.

19.1.1.6 Copies of successful demonstration of capability studies and raw data must be submitted to the Quality Assurance department.

19.1.2 A Continuing Demonstration of Capability (CDC) must be performed annually by all analysts running samples by any of the following approaches:

19.1.2.1 By using the last four of seven results obtained from a method detection limit study if run exclusively by the analyst. ONLY the last four results may be used.

19.1.2.2 BY repeating the IDC study.

19.1.2.3 By using four consecutively run blank spike results obtained during the course of routine analysis and if run exclusively by the analyst.

19.1.2.4 By running an acceptable blind performance testing sample during the course of routine sample analysis.

19.1.2.5 Copies of successful demonstration of capability studies and raw data must be submitted to the Quality Assurance department.

19.2 A Method Detection Limit (MDL) Study must be performed annually in accordance with TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8260B, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 2, December 1996

22.2 *40 Code of Federal Regulations, most current edition*, Part 136, Appendix A, Method 624-Purgeables

22.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 8260A, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 1, September, 1994

22.4 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 5030B, "Purge and Trap for Aqueous Samples", Revision 2, December, 1996

23.0 ATTACHMENTS

23.1 Attachment 23.1, Table 1, Compound List, CAS #, and Routine Reporting Limits, for Standard I

23.2 Attachment 23.2, Table 1A, Compound List, CAS #, and Routine Reporting Limits, For Standard II

23.3 Attachment 23.3, Table 2, BFB Key Ion Abundance Criteria

23.4 Attachment 23.4, Table 3, Standard 8260B Volumes Required For Curve

23.5 Attachment 23.5, Table 3A, Appendix IX Volumes Required For Curve

23.6 Attachment 23.6, Table 4, Elution Order, Quantitation and Characteristic Ions, Internal Standards, and Surrogates for Standard I

23.7 Attachment 23.7, Table 4A, Elution Order, Quantitation and Characteristic Ions, for Standard II

23.8 Attachment 23.8, Table 5, Continuing Calibration QC Acceptance Criteria, Method 624

23.9 Attachment 23.9, Example Chromatogram, DB-624

23.10 Attachment 23.10, Example Chromatogram, DB-VRX

Attachment 23.1
Table 1
Compound List, CAS Number and Routine Reporting Limits for Standard I

Mix*	Analyte	CAS Number	Concentration mg/L	Minimum Reporting Limit		
				Aqueous ug/L	Low Level Soil, mg/kg	High Level Soil, mg/kg
D	Acetone	67-64-1	100	5	0.1	0.75
C	Acrolein	107-02-8	100	5	0.01	0.05
C	Acrylonitrile	107-13-1	100	1	0.01	0.05
A	Benzene	71-43-2	100	1	0.01	0.05
A	Bromobenzene	108-86-1	100	1	0.01	0.05
A	Bromochloromethane	74-97-5	100	1	0.01	0.05
A	Bromodichloromethane	75-27-4	100	1	0.01	0.05
A	Bromoform	75-25-2	100	1	0.01	0.05
B	Bromomethane	74-83-9	100	1	0.01	0.05
A	n-Butylbenzene	104-51-8	100	1	0.01	0.05
A	sec-Butylbenzene	135-98-8	100	1	0.01	0.05
A	tert-Butylbenzene	98-06-6	100	1	0.01	0.05
D	Carbon disulfide	75-15-0	100	1	0.1	0.5
A	Carbon tetrachloride	56-23-5	100	1	0.01	0.05
A	Chlorobenzene	108-90-7	100	1	0.01	0.05
B	Chloroethane	75-00-3	100	1	0.01	0.05
D	2-Chloroethyl vinyl ether	110-75-8	100	10	0.1	0.5
A	Chloroform	67-66-3	100	1	0.01	0.05
B	Chloromethane	74-87-3	100	1	0.01	0.05
A	2-Chlorotoluene	95-49-8	100	1	0.01	0.05
A	4-Chlorotoluene	106-43-4	100	1	0.01	0.05
E	Cyclohexane	110-82-7	100	10	0.01	0.25
A	Dibromochloromethane	124-48-1	100	1	0.01	0.05
A	1,2-Dibromo-3-chloropropane	96-12-8	100	1	0.01	0.05
A	1,2-Dibromoethane	106-93-4	100	1	0.01	0.05
A	Dibromomethane	74-95-3	100	1	0.01	0.05
A	1,2-Dichlorobenzene	95-50-1	100	1	0.01	0.05
A	1,3-Dichlorobenzene	541-73-1	100	1	0.01	0.05
A	1,4-Dichlorobenzene	106-46-7	100	1	0.01	0.05
E	trans-1,4-Dichloro-2-butene	110-57-6	100	1	0.05	0.25
B	Dichlorodifluoromethane	75-71-8	100	1	0.01	0.05
A	1,1-Dichloroethane	75-34-3	100	1	0.01	0.05
A	1,2-Dichloroethane	107-06-2	100	1	0.01	0.05
A	1,1-Dichloroethylene	75-35-4	100	1	0.01	0.05
A	cis-1,2-Dichloroethylene	156-59-2	100	1	0.01	0.05
A	trans-1,2-Dichloroethylene	156-60-5	100	1	0.01	0.05
F	Dichlorofluoromethane	73-43-4	100	1	0.01	0.05
A	1,2-Dichloropropane	78-87-5	100	1	0.01	0.05
A	1,3-Dichloropropane	142-28-9	100	1	0.01	0.05
A	2,2-Dichloropropane	594-20-7	100	1	0.01	0.05
A	1,1-Dichloropropylene	563-58-6	100	1	0.01	0.05
A	cis-1,3-Dichloropropylene	10061-01-5	100	1	0.01	0.05
A	trans-1,3-Dichloropropylene	10061-02-6	100	1	0.01	0.05
A	Ethylbenzene	100-41-4	100	1	0.01	0.05
E	Ethyl ether	60-29-7	100	5	0.1	0.5
E	Heptane	142-82-5	100	10	0.1	0.5

Attachment 23.1
Table 1
Compound List, CAS Number and Routine Reporting Limits for Standard I (continued)

Mix*	Analyte	CAS Number	Concentration mg/L	Minimum Reporting Limit		
				Aqueous ug/L	Low Level Soil, mg/kg	High Level Soil, mg/kg
A	Hexachlorobutadiene	87-68-3	100	1	0.01	0.05
E	Hexachloroethane	67-72-1	100	1	0.01	0.05
D	2-Hexanone	591-78-6	100	5	0.1	2.5
D	Iodomethane	74-88-4	100	1	0.01	0.05
E	Isopropanol	67-63-0	100	25	0.1	2.5
A	Isopropylbenzene	88-82-8	100	1	0.01	0.05
A	p-Isopropyltoluene	99-87-6	100	1	0.01	0.05
E	Methyl tert-butyl ether (MTBE)	1634-04-4	100	1	0.1	2.5
E	Methyl Acetate	79-20-9	100	10	0.01	0.25
E	Methylcyclohexane	108-87-2	100	10	0.01	0.25
A	Methylene chloride	75-09-2	100	1	0.01	0.25
D	Methyl ethyl ketone (2-Butanone)	78-93-3	100	5	0.1	0.75
E	2-Methylnaphthalene	91-57-6	100	5	0.05	0.25
D	4-Methyl-2-pentanone (MIBK)	108-10-1	100	5	0.1	2.5
A	Naphthalene	91-20-3	100	5	0.01	0.5
A	n-Propylbenzene	103-65-1	100	1	0.01	0.05
A	Styrene	100-42-5	100	1	0.01	0.05
A	Tetrachloroethylene	127-18-4	100	1	0.01	0.05
A	1,1,1,2-Tetrachloroethane	630-20-6	100	1	0.01	0.05
A	1,1,2,2-Tetrachloroethane	79-34-5	100	1	0.01	0.05
A	Toluene	108-88-3	100	1	0.01	0.05
A	1,2,3-Trichlorobenzene	87-61-6	100	1	0.01	0.05
A	1,2,4-Trichlorobenzene	120-82-1	100	1	0.01	0.05
A	1,1,1-Trichloroethane	71-55-6	100	1	0.01	0.05
A	1,1,2-Trichloroethane	79-00-5	100	1	0.01	0.05
A	Trichloroethylene	79-01-6	100	1	0.01	0.05
B	Trichlorofluoromethane	75-69-4	100	1	0.01	0.05
A	1,2,3-Trichloropropane	96-18-4	100	1	0.01	0.05
E	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	100	1	0.01	0.05
A	1,2,4-Trimethylbenzene	95-63-6	100	1	0.01	0.05
A	1,3,5-Trimethylbenzene	108-67-8	100	1	0.01	0.05
D	Vinyl acetate	108-05-4	100	5	0.01	0.05
B	Vinyl chloride	75-01-4	100	1	0.01	0.05
A	m,p-Xylene	106-42-3	200	1	0.02	0.1
A	o-Xylene	95-47-6	100	1	0.01	0.05
A	Xylene, total	1330-20-7	300	1	0.03	0.15

*Mix A: Volatile Organic Compounds - Liquids @ 2.0 mg/mL in MeOH (Accustandard M502A-R-10X)

*Mix B: Volatile Organic Compounds - Gases @ 2.0 mg/mL in MeOH (Accustandard M502B-10X)

*Mix C: Acrolein & Acrylonitrile at 2.0 mg/mL in MeOH (Accustandard S-948)

*Mix D: Ketone Mix @ 2.0 mg/mL in MeOH (Accustandard M-8260-ADD-10X)

*Mix E: Custom Additions to Method 8260B @ 2.0 mg/mL in MeOH (Accustandard S-3439-R1)

*Mix F: Dichlorofluoromethane @ 1.0 mg/mL in MeOH (Absolute #70904)

Attachment 23.2
Table 1A
Compound List, CAS#, And Routine Reporting Limits For Standard II

Mix*	Analyte	CAS Number	Concentration mg/L	Minimum Reporting		
				Aqueous ug/L	Low Level Soil mg/kg	High Level Soil mg/kg
A	Allyl chloride	107-05-1	250	5	0.05	0.25
A	2-Butanol	15892-23-6	250	50	0.5	2.5
A	n-Butanol	71-36-3	250	50	0.5	2.5
A	t-Butanol	75-65-0	250	50	0.5	2.5
Neat	n-Butyl acetate	123-86-4	250	10	0.02	0.5
B	2-Chloro-1,3-butadiene (Chloroprene)	126-99-8	250	5	0.05	0.25
Neat	1-Chlorohexane	544-10-5	250	1	0.01	0.05
E	Cyclohexane	110-82-7	100	10	0.01	0.25
A	Cyclohexanone	108-94-1	250	50	0.5	2.5
A	2,3-Dichloro-1-propene	78-88-6	250	5	0.05	0.25
A	1,4-Dioxane	123-91-1	250	25	0.1	2.5
C	Epichlorohydrin	106-89-8	250	25	0.05	1.3
A	Ethanol	64-17-5	250	50	0.5	2.5
A	Ethyl acetate	141-78-6	250	10	0.5	2.5
Neat	Ethyl methacrylate	97-63-2	250	5	0.05	0.25
A	Hexachloroethane	67-72-1	250	5	0.05	0.25
A	Hexane	110-54-3	250	10	0.1	0.5
A	Isobutanol	78-83-1	250	50	0.5	2.5
A	Isobutyl acetate	110-19-0	250	5	0.1	0.5
Neat	Isopropanol	67-63-0	250	25	0.05	2.5
A	Isopropyl ether	108-20-3	250	5	0.01	0.05
A	Methacrylonitrile	126-98-7	250	5	0.5	2.5
E	Methylcyclohexane	108-87-2	100	10	0.01	0.25
Neat	Methyl methacrylate	80-62-6	250	5	0.05	0.25
A	2-Nitropropane	79-46-9	250	5	0.1	0.5
A	n-Propanol	71-23-8	250	50	0.5	2.5
A	Propionitrile	107-12-0	250	5	0.5	2.5
A	Tetrahydrofuran	109-99-9	250	5	0.1	0.5

*Mix A: Appendix IX Standard @ 2.5 mg/mL in MeOH (Accustandard S-3651 Volatile Custom Solution)
*Mix B: 2-Chloro-1,3-butadiene (Chloroprene) @ 2.0 mg/mL in MeOH (Accustandard App-9-048-R1-20X)
*Mix B: Epichlorohydrin @ 1.0 mg/mL in MeOH (Accustandard 70377)

Attachment 23.3
Table 2
BFB Key Ion Abundance Criteria

Table 2
BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

Attachment 23.4
Table 3
Standard 8260B Volumes Required For Curve

Concentration of Working Standard (ug/L)	Volume (µL) of Standard 12.4.1.1	Additional Volume (µL) of Methanol Required
1.0	0.5	999.5
5.0	2.5	997.5
10	5.0	995
20	10	990
40	20	980
100	50	950
200	100	900

Attachment 23.5
Table 3A
Appendix IX Volumes Required For Curve

Concentration of Working Standard (ug/L)	Volume (µL) of Standard 12.4.2.1	Additional Volume (µL) of Methanol Required
5.0	1.0	999
25	5.0	995
50	10	990
100	20	980
250	50	950
500	100	900
1000	20*	980

*Note: when preparing the 1000 ug/L Appendix IX calibration standard, use the stock standard listed in 12.3.4.2.

Attachment 23.6
Table 4
Elution Order, Quantitation And Characteristic Ions, Internal Standards, And Surrogates For Standard I

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard
1	Dichlodifluoromethane	85	87, 50	1
2	Chloromethane	50	52, 49	1
3	Vinyl Chloride	62	64, 47	1
4	Bromomethane	94	96, 93	1
5	Chloroethane	64	66	1
6	Trichlorofluoromethane	101	103, 105	1
7	Dichlorofluoromethane	67	69, 47	1
8	Ethyl Ether	45	59, 73	1
9	Acrolein	55	56, 53	1
10	1,1-Dichloroethylene	96	61, 98	1
11	1,1,2-Trichloro-1,2,2-trifluoroethane	101	103, 151	1
12	Iodomethane	142	127, 141	1
13	Carbon Disulfide	76	78, 77	1
14	Acetone	43	58, 42	1
17	Methylene Chloride	49	51, 84	1
18	Acrylonitrile	52	54, 53	1
19	Trans-1,2-Dichloroethylene	96	61, 98	1
20	Methyl tert-Butyl Ether (MTBE)	73	43, 57	1
21	1,1-Dichloroethane	63	65,83	1
22	Vinyl Acetate	43	42, 44	1
23	2,2-Dichloropropane	77	41, 79	1
24	cis-1,2-Dichloroethylene	96	61, 63	1
25	Methyl Ethyl Ketone (2-Butanone)	43	72, 57	1
26	Bromochloromethane	49	130, 128	1
27	Tetrahydrofuran	71	41, 42	1
28	Chloroform	83	85, 47	1
29	1,1,1-Trichloroethane	97	99, 61	1
30	SUR:Dibromofluoromethane	113	111, 192	1
31	IS:Fluorobenzene	96	70,50	1
32	Carbon Tetrachloride	117	119, 121	1
33	Cyclohexane	41	39, 57	1
34	1,1-Dichloropropylene	75	110, 77	1
35	Benzene	78	51, 50	1
36	1,2-Dichloroethane	62	64, 49	1
37	1,2-Dichloroethane-d4	65	67,51	1
38	Trichloroethylene	130	95, 132	1
39	1,2-Dichloropropane	63	62, 76	1
40	Dibromomethane	93	95, 174	1
41	Bromodichloromethane	83	85, 129	1

Attachment 23.6
Table 4
Elution Order, Quantitation And Characteristic Ions, Internal Standards, And Surrogates For Standard I (Cont.)

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard
44	cis-1,3-Dichloropropylene	75	77, 110	1
45	4-Methyl-2-Pentanone (MIBK)	43	58, 85	1
46	SUR:d8-Toluene	98	100,70	1
47	Toluene	91	92, 65	1
48	Trans-1,3-Dichloropropylene	75	77, 110	1
49	1,1,2-Trichloroethane	97	83, 85	1
50	Tetrachloroethylene	166	129, 168	2
51	1,3-Dichloropropane	76	78, 41	2
52	2-Hexanone	43	58, 85	2
53	Dibromochloromethane	129	127, 131	2
54	1,2-Dibromoethane	109	107, 188	2
55	IS:d5-Chlorobenzene	82	117, 119	2
56	Chlorobenzene	112	77, 114	2
57	1,1,1,2-Tetrachloroethane	131	133, 119	2
58	Ethylbenzene	91	106, 51	2
59	m,p-Xylene	91	106, 51	2
60	o-Xylene	91	106, 51	2
61	Styrene	104	78, 51	2
62	Bromoform	173	175, 79	2
63	Isopropylbenzene	105	120, 79	3
64	SUR:4-Bromofluorobenzene	95	174, 176	2
65	Bromobenzene	77	156, 158	3
66	1,1,2,2-Tetrachloroethane	83	85, 95	3
67	1,2,3-Trichloropropane	75	49, 110	3
68	n-Propylbenzene	91	120, 65	3
69	2-Chlorotoluene	126	91, 63	3
70	1,3,5-Trimethylbenzene	105	120, 77	3
71	4-Chlorotoluene	91	126, 63	3
72	tert-Butylbenzene	119	91, 134	3
73	sec-Butylbenzene	105	134, 91	3
74	1,3-Dichlorobenzene	146	111, 148	3
75	p-Isopropyltoluene	119	134, 91	3
76	IS:d4-1,4-Dichlorobenzene	152	150	3
77	1,4-Dichlorobenzene	146	111, 148	3
78	1,2-Dichlorobenzene	146	111, 148	3
79	n-Butylbenzene	91	92, 134	3
80	1,2-Dibromo-3-Chloropropane	75	155, 157	3
81	1,2,4-Trichlorobenzene	180	182, 145	3
82	Hexachlorobutadiene	225	227, 260	3
83	Naphthalene	128	102, 51	3
84	1,2,3-Trichlorobenzene	180	182, 145	3

Attachment 23.7
Table 4A
Elution Order, Quantitation And Characteristic Ions, For Standard II

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard
1	Ethanol	45	43	1
2	Acetonitrile	41	40, 54	1
3	Allyl Chloride	76	39, 41	1
4	Isopropanol	45	59, 43	1
5	t-Butanol	59	41, 43	1
6	Hexane	41	56, 57	1
7	Isopropylether	45	69, 86	1
8	n-Propanol	42	59, 41	1
9	Methylcyclopentane	56	41, 69	1
10	Propionitrile	54	52, 56	1
11	Ethyl Acetate	43	61, 45	1
12	Methacrylonitrile	67	41, 52	1
13	2-Butanol	45	59, 57	1
14	Sur: Dibromofluoromethane	113	111, 79	1
15	Cyclohexane	41	39, 57	1
16	IS: Fluorobenzene	96	70,50	1
17	Isobutanol	41	43, 42	1
18	n-Butanol	41	39, 56	1
19	1,2-Dichloroethane-d4	65	67,51	1
20	2,3-Dichloro-1-propylene	75	77, 110	1
21	Methyl Methacrylate	41	69, 100	1
22	1,4-Dioxane	88	57, 43	1
23	2-Nitropropane	41	43, 46	2
24	Epichlorohydrin	49	57, 62	2
25	Hexachloroethane	117	119, 201	3
26	Ethyl methacrylate	41	99, 69	2
27	Sur: d-8-Toluene	98	100, 70	2
28	n-Butyl acetate	43	56, 57	2
29	IS: d5-Chlorobenzene	82	117, 119	2
30	Cyclohexanone	55	42, 98	2
31	Sur: 4-Bromofluorobenzene	95	174, 176	2
32	trans-1,4-Dichloro-2-butylene	53	75, 89	3
33	IS: d4-1,4-Dichlorobenzene	152	150	3

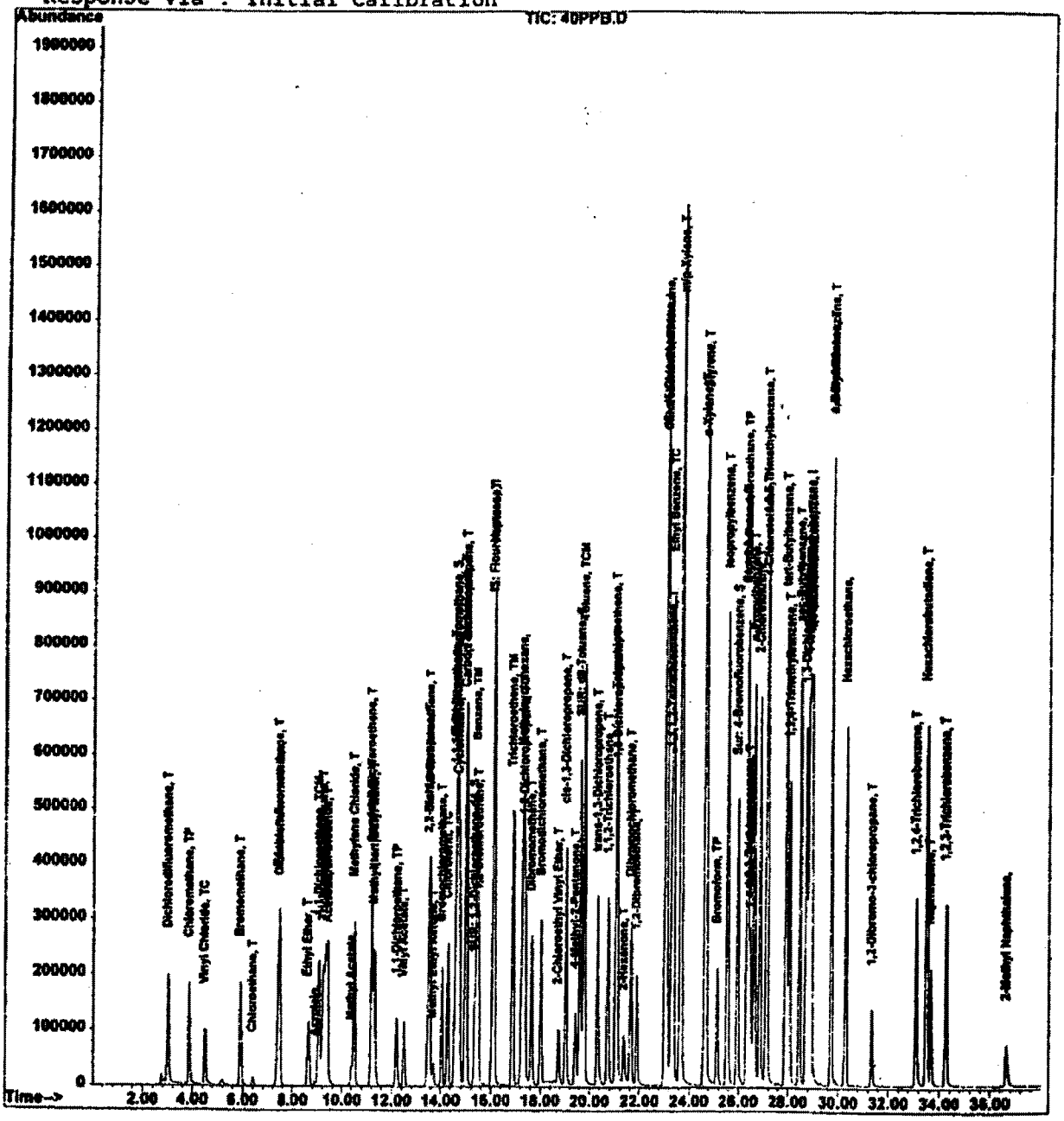
Attachment 23.8
Table 5
Continuing Calibration QC Acceptance Criteria
Method 624

Parameter	Range for Q (ug/L)
Benzene	25.6-54.4
Bromodichloromethane	26.2-53.8
Bromoform	28.4-51.6
Bromomethane	5.6-74.4
Carbon tetrachloride	29.2-50.8
Chlorobenzene	26.4-53.6
Chloroethane	15.2-64.8
2-Chloroethylvinyl ether	D-89.6
Chloroform	27.0-53.0
Chloromethane	D-81.6
Dibromochloromethane	27.0-53.0
1,2-Dichlorobenzene	25.2-54.8
1,3-Dichlorobenzene	29.2-50.8
1,4-Dichlorobenzene	25.2-54.8
1,1-Dichloroethane	29.0-51.0
1,2-Dichloroethane	27.2-52.8
1,1-Dichloroethylene	20.2-59.8
trans-1,2-Dichloroethylene	27.8-52.2
1,2-Dichloropropane	13.6-66.4
cis-1,3-Dichloropropylene	9.6-70.4
trans-1,3-Dichloropropylene	20.0-60.0
Ethyl benzene	23.6-56.4
Methylene chloride	24.2-55.8
1,1,2,2-Tetrachloroethane	24.2-55.8
Tetrachloroethylene	29.4-50.6
Toluene	29.8-50.2
1,1,1-Trichloroethane	30.0-50.0
1,1,2-Trichloroethane	28.4-51.6
Trichloroethylene	26.6-53.4
Trichlorofluoromethane	19.2-60.8
Vinyl chloride	1.6-78.4-

D=Detected result must be greater than zero.

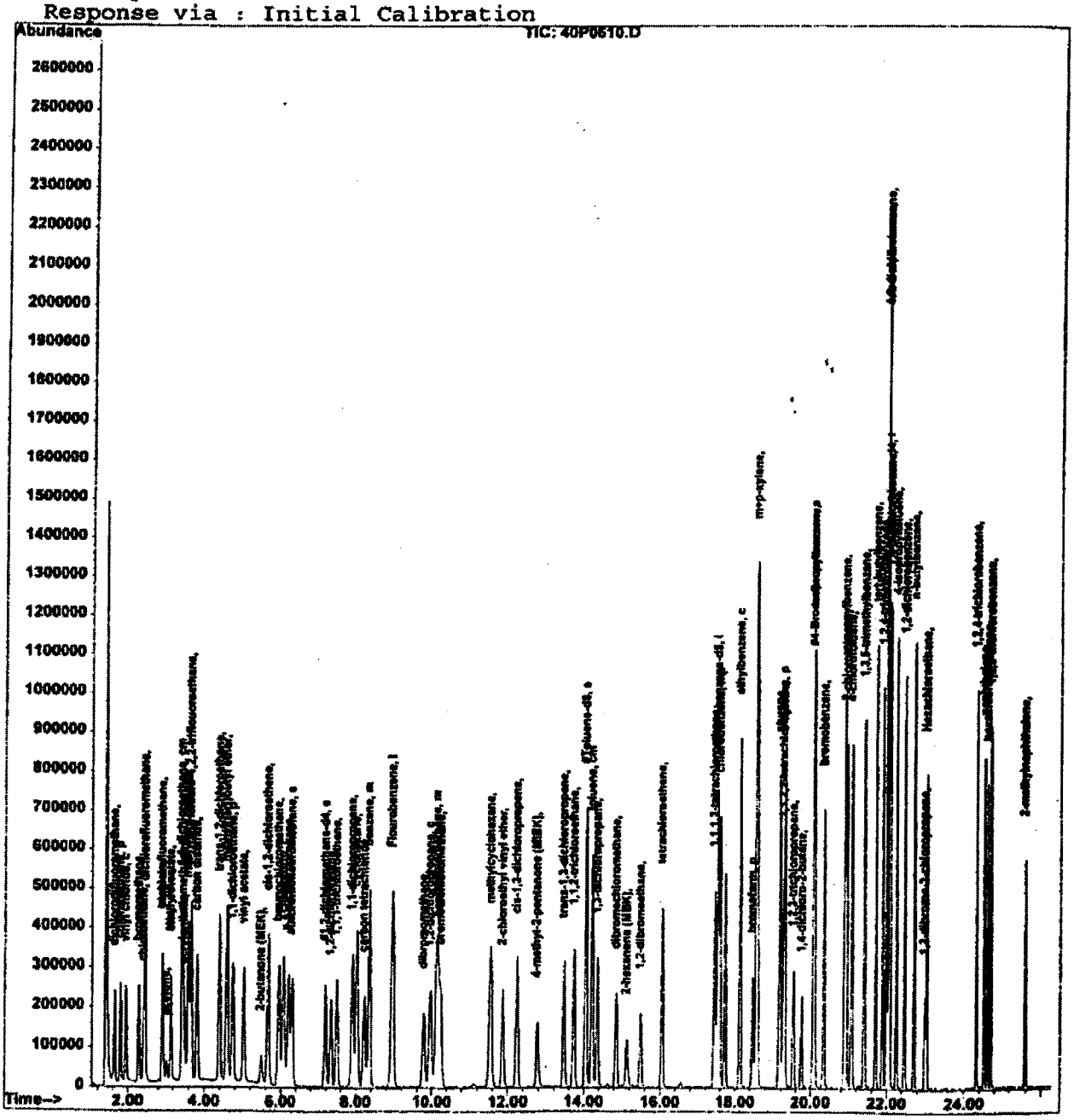
Attachment 23.9
Example Chromatogram, DB-624

```
Method        : C:\Sat132\QUANT\8260b49.m (RTE Integrator)
Title         : Saturn 132
Last Update   : Thu May 09 11:09:55 2002
Response via  : Initial Calibration
```



Attachment 23.10
Example Chromatogram, DB-VRX

```
Method        : C:\HPCHEM\2\METHODS\8260WT23.M (RTE Integrator)
Title         : EPA METHOD 624/8240
Last Update   : Thu Jun 06 15:21:31 2002
Response via  : Initial Calibration
```



TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Closed System Purge and Trap Extraction for Volatile Organic Compounds

SW-846 Method 5035A

APPROVALS:

Area Supervisor: Diane VanMale Date: 1-23-09
Diane L. VanMale

QA Officer: Tom C. Boocher Date: 1-22-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 1/26/09
Jeff P. Glaser

Procedure Number: GR-04-105
Revision Number: 1.2

Date Initiated: 4/30/98
Effective Date: 2/20/09

Date Revised: 1/22/09
Pages Revised: All

By: Diane L. VanMale
Total Number of Pages: 20

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the preparation of soils, sediments and solid waste for volatile organic compounds (VOC) analysis. The procedure includes preparation of low level, high level and oily waste.

1.2 The low level soil procedure is designed to use a hermetically-sealed 40 mL sample vial, preserved with sodium bisulfate which is sealed at the time of sample collection. Since the sample is never exposed to the atmosphere after sampling, VOC losses during shipment, storage and analysis are minimized. The low level soil procedure also details steps for collecting a sample in an appropriate storage device which can then be shipped to the laboratory where it is preserved upon receipt. The applicable concentration range for low level samples is 0.001 to 0.200 mg/kg.

1.3 The high level soil procedure is based upon preserving the sample with methanol at the time of collection or upon receipt at the laboratory. It is applicable to soil samples with VOC concentrations over 0.200 mg/kg. The closed system purge and trap employed for low concentration samples is NOT appropriate for samples preserved in the field with methanol. All methanol preserved samples must be analyzed by diluting an aliquot of the methanol into laboratory reagent water then analyzing in accordance with TriMatrix standard operating procedure (SOP) GR-04-104, GR-03-105 or GR-03-121.

1.4 This procedure can be used for most volatile organic compounds with boiling points below 200° C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, reporting limits are approximately ten times higher because of poor purging efficiency.

1.5 Refer to the appropriate analytical standard operating procedure for a target analyte list and the analysis procedure.

1.6 Low level bulk soil analysis is outside of the scope of this procedure and is no longer formally recognized by the EPA as an acceptable option for VOC analysis. However, since some states and clients still require bulk soil sample collection and analysis, the technique is included in Attachment 3.0 (Bulk Soil Collection and Preparation Technique).

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, New Methods*, Method 5035A, "Closed system Purge and trap and Extraction for Volatile Organics in Soil and Waste Samples", Draft Revision 1, July 2002

3.0 SUMMARY OF PROCEDURE

3.1 Low concentration soil procedure - Applicable to soil and other solid samples with VOC concentrations in the range of 0.001 to 0.2 mg/kg. The VOCs are determined by collecting approximately 5 g of sample and shipping it to the laboratory to be analyzed, or preserved and then analyzed. One of the following procedures must be used for sampling and preparation.

3.1.1 Collect approximately 5 g of sample by using a metal or plastic coring tool, weighed in the field at the time of collection. Place the coring in a pre-weighted 40 mL VOA vial with a septum-sealed screw cap that already contains a stir bar. The vial is then sealed and shipped to the laboratory. The sample must then be frozen within 48 hours of collection. Another option is to preserve the sample at the time of collection with sodium bisulfate preservative solution. The vial is then sealed and shipped to the laboratory where it is stored in the refrigerator until time of analysis. For analysis, the entire vial is allowed to come to room temperature, and is then placed unopened into the autosampler.

3.1.2 Alternatively, samples may be collected in the field using an appropriate coring device which can also serve as the storage device for shipping such as the EnCore™ sampler. Constructed of an inert polymer, the EnCore™ has a coring/storage chamber designed to collect a 5 g sample with a press-on cap that creates a vapor tight seal to prevent analyte loss. After collection, the sample is shipped to the laboratory where it is transferred to a new pre-cleaned and tared 40 mL VOA vial containing a stir bar. The vial is then sealed and weighed, and the weight recorded. At this point the entire vial may be placed in the freezer until time of analysis, or the appropriate volume of sodium bisulfate preservative solution (1:1 w/v) may be added, and the sample stored in the refrigerator until time of analysis. As in Section 3.1.1, the entire vial is placed unopened into the autosampler for analysis.

3.1.3 Regardless of collection technique and immediately before analysis, laboratory reagent water, surrogates and internal standards (if applicable) are automatically added without opening the sample vial. The vial is then heated to 45° C, magnetically stirred and volatiles are purged into an appropriate trap using helium gas. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped volatile analytes into a gas chromatograph (GC) for analysis by the appropriate detector.

3.2 High concentration soil procedure - Applicable to soil and other solid samples with VOC concentrations greater than 0.2 mg/kg. The low concentration technique is not applicable to all samples. Particularly those containing high concentrations of VOCs which may overload the trap or exceed the calibration range. In such instances, use one of the procedures described below.

3.2.1 The first option is to collect a bulk sample in a glass container with a PTFE-lined lid. A 5 g portion of the collected sample is removed from the container at the laboratory and added to 5.0 mL of methanol to extract volatile organic constituents. After mixing by shaking or agitating using an ultrasonic bath, a volume of the solvent extract is quantitatively diluted into laboratory reagent water and placed in a new pre-cleaned VOA vial for analysis. Surrogates and internal standards (if applicable) are added by the autosampler immediately before purging and analysis in accordance with the appropriate analytical procedure.

Note: This is the least desirable option as the sample contacts the atmosphere which may cause some volatile constituent losses.

3.2.2 The second option is to collect approximately 10 g of sample in a pre-weighed vial with a septum-sealed screw cap that contains 10 mL of methanol for extraction. For analysis, a volume of the solvent extract is quantitatively diluted into laboratory reagent water as in Section 3.2.1 and placed in a new pre-cleaned VOA vial for analysis. Surrogates and internal standards (if

applicable) are added by the autosampler immediately before purging and analysis in accordance with the appropriate analytical procedure.

3.2.3 The third option is to use a coring/storage device, such as the EnCore™ sampler described in Section 3.1.2. The EnCore™ storage chamber size can be 5 g or 25 g. Immediately after a sample is collected, seal the device and ship to the laboratory. Upon receipt, the laboratory will extrude the sample into a pre-tared 60 mL jar, weigh the sample and add methanol to the container in a 1:1 ratio of solvent volume to sample mass. At analysis, dilute an aliquot of the methanolic extract into laboratory reagent water as in Section 3.2.1 and transfer directly to a purge and trap vessel. Surrogates and internal standards (if applicable) are added by the before purging and analysis in accordance with the appropriate analytical procedure.

3.3 High concentration oily waste procedure - Applicable to oily samples with VOC concentrations greater than 0.2 mg/kg that can be diluted in a water-miscible solvent (not necessarily methanol). After demonstrating that a test aliquot is soluble in the solvent of choice, dilute a separate aliquot into the solvent. At analysis, dilute an aliquot of the water-miscible solvent extract into laboratory reagent water as in Section 3.2.1 and place in a new pre-cleaned VOA vial for analysis. Surrogates and internal standards (if applicable) are added by the autosampler immediately before purging and analysis in accordance with the appropriate analytical procedure.

Note: Oily samples not soluble in a water-miscible solvent are beyond the scope of this procedure and must be analyzed by direct injection analysis after dilution in n-hexadecane.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to each analytical procedure for the appropriate parameters list.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-04-104, *Volatile Organic Compounds By Purge And Trap Capillary Column Gas Chromatography/Mass Spectrometry*, latest revision

5.2 TriMatrix SOP GR-03-105, *Volatile Organic Compounds By Purge And Trap Capillary Column Gas Chromatography With Photoionization And Electrolytic Conductivity Detectors in Series*, latest revision

5.3 TriMatrix SOP GR-03-121, *Method for the Determination of Gasoline Range Organics*, latest revision

5.4 TriMatrix SOP GR-03-124, *Volatile Organic Laboratory Corrective Actions*, latest revision

5.5 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.6 TriMatrix SOP GR-10-113, *Laboratory Balance Calibration and Verification*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Sample contamination can cause elevate reporting limits or give false positives and come from a variety of sources. Improper sampling can contaminate samples at the job site. During shipment and storage, volatile organics (particularly methylene chloride and fluorocarbons) can diffuse through the septum. A trip blank prepared from laboratory reagent water containing the sodium bisulfate preservative and carried through all sampling and handling serves as a check on contamination for the low level procedure. For the methanol preservation technique, a trip blank must be prepared using purge and trap grade methanol which must then be processed through all sample preparation and analysis steps.

6.2 To minimize contamination, the volatiles analysis laboratory must be free of solvents. The volatiles sample storage area must be isolated from atmospheric sources of methylene chloride and other solvents. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing must be stainless steel or copper. Clothing previously exposed to methylene chloride fumes during semi-volatiles extraction can contribute to sample contamination and must not be worn into the volatiles laboratory.

6.3 The sodium bisulfate preservative acts to lower sample pH which inhibits biological degradation of aromatic compounds. However, under acidic conditions, highly reactive compounds such as 2-Chloroethylvinyl ether are unstable and will be lost. Acid preservation can also degrade methyl-t-butyl ether (MtBE) to tert-butyl alcohol during purge and trap. Additional analytes affected include vinyl chloride, styrene and other fuel oxygenate ethers. If these analytes are to be reported, collect a second sample set without sodium bisulfate preservative. Acidification of certain soil types with sodium bisulfate can produce an acetone artifact which is typically between 0.025 and 0.100 mg/kg.

6.4 For the appropriate corrective actions when encountering contamination, refer to TriMatrix SOP GR-03-124.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 The toxicity or carcinogenicity of reagents used in this procedure has not been precisely defined. However, each chemical must be treated as a potential health hazard and exposure must be reduced to the lowest possible level by all means available.

7.3 The laboratory maintains a current reference file of material safety data sheets (MSDS) available to all personnel.

7.4 Many of the analytes tested have been classified as known or suspected carcinogens. Consequently, all neat standard materials and standard solutions must be opened only in a fume hood.

7.5 Appropriate personal protective equipment including a laboratory coat, approved safety glasses and disposable gloves must be worn in the laboratory when handling any chemical. The only exception for wearing personal protective equipment is when working at a computer station in the laboratory.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 The specific sample container required depends on the purge and trap system. TriMatrix Laboratories currently uses the needle sparge type purging device for all low level soils. This system requires a 40 mL borosilicate glass volatile organic analysis (VOA) vial.

8.2 The specific preparation for a sample vial depends on the expected concentration range with specific preparation techniques for low concentration soils, high concentration soils and solid waste. Sample vials must be prepared in the laboratory or other controlled environment then sealed and shipped to the collection site. Disposable gloves must be worn during the preparation steps which are as follows:

8.2.1 The following instructions apply to the preparation of vials used in the collection of low concentration soil samples that are to be preserved in the field.

8.2.1.1 Add a clean magnetic stirring bar to each clean vial.

8.2.1.2 Add 5.0 mL of 20% sodium bisulfate preservative solution to each vial. This should be sufficient to ensure a sample pH of <2 to prevent biodegradation.

8.2.1.3 Seal the vial with the screw-cap septum lid.

8.2.1.4 Affix a label to each vial. This eliminates the need to label vials in the field and assures that the vial tare weight is included on the label.

8.2.1.5 Weigh the prepared vial to the nearest 0.01 g, record the tare weight and write it on the label.

8.2.1.6 Because volatile organics will partition into the vial headspace from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes and internal standards (if applicable) must only be added after sample has been added. Surrogates, matrix spikes and internal standards (if applicable) must be injected automatically by the autosampler just prior to analysis.

8.2.2 When high concentration samples are collected without a preservative, a variety of sample containers may be employed including 40, 60 and 125 mL glass jar with a PTFE liner. This collection technique must only be used when sample solubility in methanol is questionable.

8.2.3 The following steps apply for high concentration soil samples collected and preserved in the field with methanol as described in Section 3.2.

8.2.3.1 Use pre-tared 40, 60 or 125 mL jars with screw cap lids and PFTE liners.

8.2.3.2 To obtain the pre-tared weighing, weigh the container with the label, lid and liner in place then record the tare weight on the label to the nearest 0.01 g. The mass of the ink is negligible.

8.2.3.3 Include a purchased 10 mL ampule of purge and trap methanol with each sample container.

8.2.4 When oily waste samples are known to be soluble in methanol, sample containers may be used as described in Section 8.2.3, preserving with methanol. However, when methanol solubility is unknown, collect the sample without preservative in a 40, 60 or 125 mL glass jar with a PTFE liner.

8.2.5 Preservation by freezing to less than -7° C may be used unless otherwise specified and is as follows:

8.2.5.1 If freezing at collection, add sample to an empty sample container and freeze to less than -7° C until analysis.

Note: When freezing, always place the sample container on its side to prevent the glass container from breaking.

8.2.5.2 Samples received by the laboratory unfrozen and at 4 ±2° C, and are within 48 hours of collection may be frozen to less than -7° C at the time of receipt.

8.2.5.3 Samples must be thawed for preparation and analysis within 14 days of collection.

8.2.5.4 Thawed samples must be prepared and analyzed within 24 hours of thawing.

8.3 Sample Collection

8.3.1 Collect samples according to the procedure outlined in the project sampling plan. In general, after a fresh surface of the solid material is exposed, collect a subsample in the least amount of time and with as little disruption as possible to minimize the loss of volatiles. Use a metal or rigid plastic coring tool to collect the subsample. These devices help maintain sample structure during collection and transfer to the sample container. When inserting the coring device into solid material, use care not to trap air behind the sample. If air is trapped, it can cause VOC loss by passing through the sample or cause the sample to be pushed from coring device before a transfer can be made to the sample container. The following devices have been designed to prevent headspace during collection and are approved for sampling:

8.3.1.1 EasyDraw Syringe™ with Powerstop Handle™

8.3.1.2 Terra Core™

8.3.1.3 EnCore™

8.3.2 Low level soil samples may be collected as follows:

8.3.2.1 Collect approximately 5 g of soil using a coring device as described in Section 8.3.1. After taking the sample, remove the filled corer and quickly wipe the barrel with a clean disposable wipe.

8.3.2.2 If a sample is to be preserved at collection, add to a sample jar containing sodium bisulfate preservative by holding the sample jar at an angle then extrude into the

container to minimize splashing. Quickly brush or wipe the vial threads to remove residual soil and immediately seal the vial tightly with the septum screw cap.

Note: An improper seal due to soil remaining on the sample jar threads and improper lid tightening are the primary factors in volatiles loss during sample collection. This can also cause problems during analysis by preventing the vial from pressurizing during the purge step, resulting in low or no recovery.

8.3.2.3 If a sample is shipped to the laboratory for preserving, cap the open end of the coring/storage device after ensuring all sealing surfaces are clean then store and transport at 4 ±2° C.

Note: An individual EnCore™ is needed for each sample aliquot.

8.3.2.4 If samples are expected to contain target analytes over a wide concentration range and high level samples are not being collected, collect an additional sample aliquot in a low concentration preserved VOA vial with 1 - 2 g rather than 5 g. If necessary, the low mass sample can be analyzed for analytes in the 5 g sample that exceed the calibration range.

8.3.2.5 Samples that contain carbonate minerals may effervesce upon contact with the sodium bisulfate preservative. If the gas volume generated is small, volatiles loss may be minimal. However, if large gas volumes are generated, volatiles are likely to be lost and the vial may rupture. If carbonate minerals are expected, collect a test sample to check for effervescence. If the sample effervesces when sodium bisulfate is added, discard the test sample and collect additional sample without preserving.

8.3.3 High level soil samples may be collected as follows:

8.3.3.1 Preserve in the field as follows:

8.3.3.1.1 Use a coring device to collect a 5 g or 25 g sample as soon as possible after solid surface material is exposed. Wipe the exterior of the collection device with a clean disposable wipe.

8.3.3.1.2 Quickly transfer to a 40 mL vial for 5 g, or to a 60 or 125 mL sample jar for a 25 g sample.

8.3.3.1.3 Add a volume of methanol in a 1:1 ratio of methanol volume to sample mass.

8.3.3.2 Preserved in the laboratory as follows:

8.3.3.2.1 If a sample is to be preserved in the laboratory, collect in an EnCore™ sampler, cap the open end after ensuring all sealing surfaces are clean and store at 4 ±2° C until transport.

8.3.3.2.2 Upon receipt at the laboratory, the sample must be extruded from the EnCore™ into a new sample jar then weighed and a 1:1 ratio of methanol volume to sample mass added. Refer to Section 13.0 for detailed instructions.

8.4 Sample Storage

8.4.1 Once in the laboratory, store samples at 4 ±2° C (or freeze to -7° C within 48 hours of collection) until analysis. The volatiles sample storage area must be free of organic solvent vapors.

8.4.2 All samples must be analyzed as soon as practical and within the designated holding time. Samples not analyzed within the holding time must be reported as exceeding the holding time. Results must be reported as estimated minimum values.

8.4.4 When low concentration samples are strongly alkaline or highly calcareous, the sodium bisulfate preservative solution may not be strong enough to reduce pH to below 2.

8.4.4.1 When low concentration soils are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required for preservation. Such steps include:

8.4.4.1.1 Addition of a larger sodium bisulfate preservative volume

8.4.4.1.2 Storage at below -10° C (taking care not to fill the vial it breaks from expansion)

8.4.4.1.3 Significantly reducing the maximum holding time.

8.4.4.2 The preservation used must be clearly described in the sampling and quality assurance (QA) project plan for distribution to both field and laboratory personnel.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Sample containers are as follows:

9.1.1 Vials, 20 or 40 mL with PTFE-lined septum-sealed screw cap lids

Note: Examine each vial prior to use to ensure the vial has a flat, uniform sealing surface.

9.1.2 Glass borosilicate jars, 60 or 125 mL with PTFE-lined screw cap lids

9.2 EnCore™ sampler (EnNovative Technologies, Inc.)

9.3 microSyringes, 100 µL and 1000 µL

9.4 Balance, top-loading, capable of accurately weighing to 0.01 g

9.5 Disposable Pasteur pipets

9.6 Magnetic stirring bars, PTFE of the appropriate sizes to fit sample containers.

Note: Stirring bars may be reused provided they are thoroughly cleaned between each use.

9.7 Field equipment is as follows:

9.7.1 Terra Core™ sampler, EnNovative Technologies, Inc.

9.7.2 EasyDraw Syringe™ with Powerstop Handle™, US Oil Company

9.7.3 Portable balance, capable of accurately weighing to 0.01g

9.8 Volumetric flask, 500 mL

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no routine preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, organic-free

11.2 Methanol (CH_3OH), purge and trap grade

Note: Store away from other solvents

11.3 The low concentration sample preservative is prepared as follows:

11.3.1 Sodium bisulfate ($NaHSO_4$), certified

11.3.2 Weigh 100 g of $NaHSO_4$ into a 500 mL volumetric flask. Mix to dissolve the $NaHSO_4$ then bring to volume with laboratory reagent water (organic-free). Record the preparation in the laboratory information system (Element™) and/or in the reagent preparation logbook. Concentration is 20% (w/v) $NaHSO_4$.

11.3.3 Expiration is 6 months from the date prepared.

11.4 Refer to the appropriate analytical procedure for guidance on preparing internal standards and/or surrogates.

12.0 STANDARDS PREPARATION

12.1 There are no analytical standards directly associated with this procedure.

13.0 SAMPLE PREPARATION

13.1 Prepare low level soils collected with an Encore sampler (5 g) as follows:

13.1.1 Place a 40 mL vial on the top-loading balance and tare with the cap off.

13.1.2 Empty the 5 g Encore sample into the vial.

13.1.3 Weigh to the nearest 0.01 g and record on the vial label.

13.1.4 Add the equivalent volume of 20% $NaHSO_4$ to the vial to achieve a 1:1 ratio of sample mass to preservative volume.

13.1.5 Add a clean magnetic stir bar to the vial and seal the vial.

13.1.6 Remember to attach the label to the vial.

Note: Sodium bisulfate degrades 2-chloroethylvinyl ether. TriMatrix Laboratories has demonstrated that this preservative will not give acceptable quality control recovery so 2-chloroethylvinyl ether will not be reported when used.

13.2 Prepare high level soils collected with an Encore sampler (25 g) as follows:

13.2.1 Place 60 mL sample jar on the top-loading balance and tare with the cap off.

13.2.2 Empty the 25 g Encore sample into the jar

13.2.3 Weigh to the nearest 0.01 g and record on the jar label.

13.2.4 Add the equivalent volume of methanol to the vial to achieve a 1:1 ratio of sample mass to preservative volume then seal the jar.

13.2.6 Remember to attach the label to the jar.

13.2.7 Shake the sample jar for 2 minutes. Then sonicate the jar for 20 minutes.

13.2.8 After shaking and sonicating, place in a refrigerator at 4 ±2° C for one hour to let settle.

13.2.9 After settling, carefully decant enough methanol extract to fill a 4 mL vial without headspace and seal with the septum screw cap. Store at 4 ±2° C until analysis.

13.3 Prepare high level soils pre-preserved in the field as follows:

13.3.1 Weigh the sample jar containing soil sample and methanol, and record on the label.

13.3.2 Enter the sample jar size and mass into the Volatile Soil Receipt Information spreadsheet located on the laboratory intranet library (Attachments 1 and 2).

13.3.4 The spreadsheet determines if additional methanol needs added and/or whether the sample can be used, as follows:

13.3.4.1 If the mass collected in ratio with the methanol added is less than a 1:1 ratio of sample to preservative, consult the project chemist. The sample must be reported with a low weight narration.

13.3.4.2 If the mass collected requires additional methanol that exceeds the sample jar volume, consult the project chemist. The sample may be rejected for analysis.

13.3.4.3 If the mass collected in ratio with the methanol added is greater than a 1:1 ratio of sample to preservative, add the appropriate methanol volume.

13.3.5 Shake the sample jar for 2 minutes. Then sonicate the sample jar for 20 minutes.

13.3.6 After shaking and sonicating, place in a refrigerator at 4 ±2° C for one hour to let settle.

13.3.7 After settling, carefully decant enough methanol extract to fill a 4 mL vial without headspace and seal with the septum screw cap. Store at 4 ±2° C until analysis.

13.4 Prepare oily waste samples soluble in a water-miscible solvent as follows:

13.4.1 Place a 20 mL vial on the top-loading balance and tare with the cap off.

13.4.2 Weigh out 1 g of sample to the nearest 0.01 g.

13.4.3 Add 10 mL of water-miscible solvent and quickly cap the vial.

13.4.4 Shake for 2 minutes. After shaking or sonicating, place in a refrigerator at 4 ±2° C for one hour to let settle.

13.4.5 After settling, carefully decant enough of the mixture to fill a 4 mL vial without headspace and seal with the septum screw cap. A 1:50 dilution in the water-miscible solvent will be made unless the analyst suspects higher volatile compound concentrations.

Note: Oily samples that are not soluble in a water-miscible solvent are beyond the scope of this procedure.

14.0 CALIBRATION PROCEDURES

14.1 There are no calibration procedures directly associated with this procedure.

15.0 ANALYTICAL PROCEDURE

15.1 Analyze a low level soil as follows:

15.1.1 Remove the sample vial from storage and bring to room temperature. Shake the vial gently, to ensure the contents move freely and that stirring will be effective. Place the sample vial in the autosampler.

15.1.2 For matrix spiked samples, add the matrix spiking solution described in the appropriate analytical procedure. The spiking solution concentration and the volume injected will vary.

15.1.3 Perform the qualitative and quantitative analysis in accordance with every step in the analytical procedure.

15.1.4 If any target analyte concentration exceeds the calibration range, reanalyze by the high concentration procedure. Reanalysis need only address analytes exceeding the calibration range.

15.1.5 Alternatively, analyze the 1-2 g sample aliquot if collected (Refer to Section 8.2.2.7). Reanalysis need only address analytes exceeding the calibration range in the 5 g sample analysis.

15.2 Analyze a high level soil or waste as follows:

15.2.1 Pipet 1.0 mL of solvent extract or diluant to a 50 mL volumetric flask approximately ¾ full of laboratory reagent water (organic-free). Dilute to volume with laboratory reagent water organic-free) and invert 3 times to mix. Transfer to a 40 mL vial and place on the autosampler for analysis.

15.2.2 For matrix spiked samples, add the matrix spiking solution described in the appropriate analytical procedure. The spiking solution concentration and the volume injected will vary.

15.2.3 Perform the qualitative and quantitative analysis in accordance with every step in the analytical procedure.

15.2.4 If any target analyte concentration exceeds the calibration range, reanalyze by diluting a lesser amount of the solvent extract into a 50 ml volumetric flask. Reanalysis need only address analytes exceeding the calibration range.

16.0 CALCULATIONS AND DATA HANDLING

16.1 There are no analytical calculations directly associated with this procedure.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Volatiles Soil Receipt Information Spreadsheets are to be archived as raw data.

17.2 Refer to the analytical procedure for complete data reporting and deliverable requirements.

18.0 QUALITY ASSURANCE

18.1 Refer to the analytical procedure for complete quality control/quality assurance requirements.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Refer to the analytical procedure for demonstration of capability study and instrument validation requirements.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, New Methods*, Method 5035A, "Closed system Purge and trap and Extraction for Volatile Organics in Soil and Waste Samples", Draft Revision 1, July 2002

23.0 ATTACHMENTS

23.1 Volatile Soil Sample Receipt Information Spreadsheet, Page 1

23.2 Volatile Soil Sample Receipt Information Spreadsheet, Page 1

23.3 Bulk Soil Collection and Preparation

Attachment 23.1
Volatile Soil Sample Receipt Information Spreadsheet, Page 1

TriMatrix Laboratories, Inc.

Volatile Soil Sample Receipt Information Sheet

Client: _____ Date Received: 08/29/2003 Sheet Completed By: JDM

Project-Submittal: _____ Date Form Completed: 09/03/2003 Sheet Reviewed By: _____

Low Level Soils	Yes	No	N/A
Were Samples Received in 40 mL VOA Vials Containing a Stir Bar and Pre-Preserved with Sodium Bisulfate?	☐	☐	☐
Were Samples Received Non-Preserved in Encore Samplers?	☐	☐	☐
If Received in Encore Samplers, Was Sample Received Within 48 Hours?	☐	☐	☐
Volatile Lab Informed That Samples Must Be Preserved AND Analyzed Within 48 Hours?	☐	☐	☐

High Level Soils	Yes	No	N/A
Extra Sample Containers Received for Dry Weight Determination?	☐	☐	☐
MeOH Trip Blank Received (if MeOH not added from sealed ampules)?	☐	☐	☐
Samples Collected in Which of the Following Ways?			
Tared 40, 60, or 120 mL Containers:			
Were Containers Supplied by TriMatrix?	☐	☐	☐
Were Samples Received Within 4 Days After Collection, and Were They MeOH Preserved?	☐	☐	☐
Packed With No Headspace into Brass Tubes:			
Were Samples Received Within 4 Days After Collection, and Preserved Within 2 Hours of Sample Receipt?	☐	☐	☐
Packed With No Headspace in En Core Samplers:			
Were Samples Received Within 40 Hours After Collection and Preserved Within 48 Hours of Collection?	☐	☐	☐

Follow-Up	Yes	No	N/A
Has Soil Weight Table Been Completed?	☐	☐	☐
Has Project Chemist Been Informed of any Out-of-Compliance Samples?	☐	☐	☐

0903C.xls page #: 1 of 2 revision 0.0

Approved By: MD 1-22-09 **QA Officer**

Approved By: DW 1-23-09 **Area Supervisor**

Attachment 23.2
Volatile Soil Sample Receipt Information Spreadsheet, Page 2

TriMatrix Laboratories, Inc.

Volatile Soil Sample Receipt Information Sheet

Client: _____ Date Received: 08/29/2003 Sheet Completed By: JDM

Project-Submittal: _____ Date Form Completed: 09/03/2003 Sheet Reviewed By: _____

Client Sample Identification	TriMatrix Sample Number	Vial Size: 40, 60, or 120 mL	Empty Weight of Bottle (g)	MeOH Added: 10, 25, or 50 mL	Full Weight of Bottle (g)	Date Sample Weighed	Time Sample Weighed	Weight of Soil (g)	Low Wt. Flag Required?	Additional MeOH Required?	Accept/ Reject Sample	MeOH to Add (mL)	mL MeOH Added: By, Date, Time
1	341527	40	25.5	10	43.6			10.2			Accept Sample		
2	341528	40	25.6	10	43.6			10.1			Accept Sample		
3	341543	40	25.6	10	43.4			9.9			Accept Sample		
4	341544	40	25.3	10	43.0			9.8			Accept Sample		
5	341546	40	25.6	10	43.7			10.2			Accept Sample		
6	341547	40	25.3	10	43.3			10.1			Accept Sample		
7	341548	40	25.3	10	43.3			10.1			Accept Sample		
8	341549	40	25.3	10	43.4			10.2			Accept Sample		
9	341550	40	25.4	10	43.9			10.6			Accept Sample		

0903C.xls page #: 2 of 2 revision 0.0

Approved By: JDJ 1-22-09 QA Officer

Approved By: DWJ 1-23-09 Area Supervisor

Attachment 23.3
Bulk Soil Collection and Preparation

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the collection and preparation of bulk soils and sediments for volatile organic compounds (VOC) analysis. Low level bulk soil analysis is no longer formally recognized by the EPA as an acceptable option for VOCs. However, some states and/or clients still request bulk soil sample collection and analysis.

2.0 SUMMARY OF PROCEDURE

2.1 Bulk soil samples are collected with minimal headspace in either 60 or 125 mL glass jars. After collection, samples must be stored at 4 ±°2 C until transport to the laboratory.

2.2 When received by the laboratory, samples remain stored at 4 ±°2 C until the analyst transfers a 1-5 g subsample to a new 40 mL vial containing a stir bar for preparation.

2.3 The vial is then loaded onto the autosampler for analysis.

3.0 INTERFERENCES AND CORRECTIVE PROCEDURES

3.1 Bulk soil sampling is inaccurate and produces results that are biased low. Bulk samples can lose more than 90% VOC content prior to analytical measurement.

3.2 Reasons for such losses include the following:

3.2.1 Volatilization from exposure of the solid surface near the time of collection

3.2.2 Volatilization from intermediate storage containers

3.2.3. Volatilization from the disaggregation of the solid material during collection

3.2.4 Volatilization from failed seals on sample jar lids

3.2.5 Volatilization during laboratory subsampling

3.2.6 Biodegradation (primarily aromatic compounds) during storage

3.2.7 Chemical reactions during storage

4.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

Attachment 23.3 (continued)
Bulk Soil Collection and Preparation

4.1 Solid samples are collected using a stainless steel spatula-type device to completely fill a 40, 60 or 125 mL glass jar.

4.2 The sample container is then closed using PTFE-lined caps and stored at 4 ±2° C with ice throughout the collection event, during transport to the laboratory and during storage at the laboratory until just prior to analysis.

4.3 The holding time for samples is 14 days from the time of collection.

4.4 If samples require high level preparation/analysis based on low level results or the sample matrix, a subsample must be extracted using methanol.

4.5 The holding time for a methanol extract is also 14 days from the time of collection.

5.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

5.1 Refer to Section 9.0 in the main body of the procedure.

5.0 SAMPLE PREPARATION

6.1 Prepare a low level sample as follows:

6.1.1 From the original sample container, remove a representative subsample and transfer to a tared 40 mL vial containing a PTFE stir bar. Record the weight to the nearest 0.01 g. Use a 5 g sample unless higher level concentrations are expected and a smaller aliquot is needed.

Note: Do not use less than a 1 g sample aliquot.

6.1.2 Quickly wipe any residual soil from the vial threads and seal then load onto the instrument autosampler. The autosampler will add 10 mL of laboratory reagent water (organic-free) and applicable internal standards and/or surrogates immediately prior to the purge cycle.

6.2 Prepare a high level sample as follows:

6.2.1 If low level analysis results are outside the instrument calibration range or if the sample matrix indicates an unsuitability for low level analysis, the sample requires high level preparation and analysis.

6.2.2 From the original sample jar, remove a representative subsample and transfer to a tared 20 mL sample vial. Weigh out 5 g to the nearest 0.1 g and record on the label. Remove any residual

Attachment 23.3 (continued)
Bulk Soil Collection and Preparation

sample from the vial threads and pipet in 5.0 mL of methanol. Cap and shake the container by hand for 2 minutes. Place in the refrigerator at 4 ±2° C to settle out.

Note: Some samples may require letting stand overnight for sufficient phase separation.

6.2.3 Once the phases separate, decant the methanol phase into a new 4 mL vial for storage. Store at 4 ±2° C until analysis.

Note: The methanol must be withdrawn from the sample within 24 hours of extracting the sample to keep the extraction times constant.

7.0 CHEMICALS AND REAGENTS

7.1 Refer to Section 11.0 in the main body of the procedure.

8.0 ANALYTICAL PROCEDURE

8.1 Once prepared, low level soils can be directly loaded onto the purge and trap autosampler.

8.2 The autosampler will add laboratory reagent water (organic-free), internal standards and surrogates.

8.3 If the results of the analysis are outside the calibration range, reanalyze as a high level sample as follows:

8.2.1.1 Pipet 1.0 mL of extract into a 50 mL volumetric flask approximately ¾ full of laboratory reagent water (organic-free).

8.2.1.2 Dilute to volume with laboratory reagent water (organic-free).

8.2.1.3 Transfer to a 40 mL vial and place vial on autosampler for analysis.

8.4 If analysis of the high level preparation exceeds the calibration range, reanalyze by using an appropriate smaller extract volume to dilute into the 50 mL volumetric flask or perform a serial dilution until response is with the calibration range.

8.5 Follow the main body of the procedure to complete analysis and data reduction.

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Extraction of Base Neutrals and Acids from Water

EPA Method 625
SW-846 Method 3510C

APPROVALS:

Area Supervisor: Brian Hall — Brian J. Hall — Date: 2/12/09

QA Officer: _____ — Tom C. Boocher — Date: 2-12-09

Operations Manager: Jeff P. Glaser — Jeff P. Glaser — Date: 2/12/09

Procedure Number: GR-09-101
Revision Number: 3.3

Date Initiated: 1/18/96
Effective Date: 3/12/09

Date Revised: 2/12/09
Pages Revised: All

By: Dan J. Mierendorf
Total Number of Pages: 22

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure describes extraction of insoluble and slightly soluble semi-volatile organic base-neutral and acid compounds from aqueous samples and TCLP leachates. Also described are concentration techniques preparatory to GC/MS analysis.

1.2 The procedure is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results by performance of a satisfactory demonstration of capability study.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition.* Final Update III, Revision 3, December 1996, Method 3510C, "Separatory Funnel Liquid-Liquid Extraction"

2.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids

3.0 SUMMARY OF PROCEDURE

3.1 A measured sample volume, usually 1 L, is serially extracted with methylene chloride at acidic pH, then again at basic pH using separatory funnels.

3.2 Extracts are dried and concentrated to a final volume of 1.0 mL or 10 mL before GCMS analysis.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to Table 1 in TriMatrix SOP GR-04-103 for a compound list associated with this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-04-103, *Semi-Volatile Laboratory Ion Trap Mass Spectrometry Analysis of Base/Neutral/Acid Compounds*, latest revision

5.2 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration*, latest revision

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.4 TriMatrix SOP GR-16-100, *Organic Glassware Cleaning,* latest revision

5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interferences can be caused by contaminants in solvents, reagents, glassware or sample processing hardware. Contamination leads to discrete artifacts and elevated baselines. A method performance blank (MPB) must be performed routinely, to demonstrate that equipment and reagents are not contaminated.

6.2 Always use high purity reagents, solvents and clean glassware, cleaned in accordance with TriMatrix SOP GR-16-100.

6.3 Matrix interference is co-extracted with analyte from samples. Extraction interferences will vary considerably from matrix to matrix.

6.4 Phthalate esters co-elute during analysis as extraneous chromatographic peaks. Do not use any type of flexible plastic during extraction. Contact with extraction solvents will leach phthalate esters into sample extracts, presenting interference in compound identification and quantitation.

6.5 Matrix interference can also affect analyte recovery. Re-extraction may be necessary if surrogates or other spiked compounds fail established laboratory recovery limits.

7.0 SAFETY PRECAUTIONS

7.1 Extraction personnel must wear lab coats and approved safety glasses while in the prep lab area. In addition, disposable elastic gloves must be worn whenever samples, solvents or reagents are handled.

7.2 For extractions and surrogate/spike/reagent solution preparations, follow all safety instructions outlined in the latest revision of the Chemical Hygiene Plan and Laboratory Safety Manual.

7.3 For proper spill response, chemical storage and waste disposal refer to the latest revision of TriMatrix SOP GR-15-102.

7.4 Toxicity and carcinogenicity of materials used in this procedure have not been completely and precisely defined.

7.4.1 All chemicals must be treated as a potential health hazard.

7.4.2 Exposure can be reduced to the lowest possible level by adherence to established safety practices.

7.4.3 Material Safety Data Sheets are maintained within the laboratory (in the intranet library) of all chemicals used in this procedure.

7.5 Safety issues must be brought to the immediate attention of the area supervisor.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are collected in 1000 mL screw-cap amber glass jars with PTFE-lined lids. No preservative is required.

8.2 All aqueous samples must be extracted within 7 days of the collection date. TCLP leachates must be extracted within 7 days of complete leachate generation. Extract analysis must occur within 40 days of the extraction.

8.3 When not in use, samples must be stored in the walk-in cooler at 4° C ± 2° C, or as specified by the project.

8.4 Analysts must use care when handling sample containers to avoid loss or injury due to breakage.

8.5 After extraction, extracts must be stored in the GC/MS refrigerator at 4° C until analysis.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Graduated cylinders: 10 mL, 100 mL, 1000 mL

9.2 Separatory funnels with PTFE stopcocks: 2 L

9.3 Erlenmeyer flasks: 250 mL, 500 mL

9.4 Kuderna-Danish (K-D) concentrator glassware

9.4.1 Concentrator tubes: 10 mL, graduated (Kontes K-570050-1025 or equivalent)

9.4.2 Concentrator flasks: 500 mL (Kontes K-570001-500 or equivalent)

9.4.3 Concentrator tube clips

9.4.4 Snyder columns: Three-ball macro (Kontes K-50300-0121 or equivalent)

9.4.5 Snyder columns: Two-ball micro

9.5 pH indicator strips: pH range 0-14

9.6 Filter funnels: 100 mm

9.7 Filter funnels: 250 mL, ribbed

9.8 Filter paper: Fisher P8 qualitative or equivalent

9.9 Glass wool

9.10 Pasteur pipettes: Glass disposable, 2 mL

9.11 Water bath: Capable of variable temperature control to ±5° C

9.12 Hot plate: Capable of variable temperature control to ±5° C

9.13 Beakers, glass: 100 mL, 400 mL and 800 mL

9.14 Boiling chips: PTFE, rinsed with methylene chloride

9.15 Volumetric Flasks: 10 mL, 50 mL, 100 mL, 1 L

9.16 Vials with PTFE-lined screw-cap lids: Amber, 40 mL

9.17 Syringes, micro gastight: 100 uL and 1000 uL

9.18 Centrifuge tubes: PTFE, 40 mL

9.19 Centrifuge: Variable speed

9.20 Vials: Glass auto-sampler with PTFE-lined screw-cap lids, 1.7 mL calibrated to 1.0 mL (as outlined in SOP number GR-09-106)

9.21 Vials: Glass PTFE-lined screw-cap lids, 14 mL calibrated to 10 mL (refer to TriMatrix SOP GR-09-106)

9.22 Balance: Analytical, capable of accurate measurement to the nearest 0.0001 g

9.23 Sodium sulfate rinsing equipment

9.23.1 Buchner funnel: 20 cm

9.23.2 Flask: 4 L Vacuum

9.23.3 Drying pan: 13 x 9 inch, metal

9.23.4 Vacuum pump

9.23.5 Drying oven

9.23.6 Stopper: PTFE, with hole for the Buchner funnel

9.23.7 Clamps: supporting

9.23.8 Filter paper: qualitative fast, 20 cm

9.23.9 Squirt bottle: PTFE only

9.24 Phase separation paper (JT Baker 8002-15)

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Glass syringes must be cleaned or replaced when needles appear bent or plugged.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water

11.1.1 Reagent water is laboratory-purified and methylene chloride extracted (unless otherwise specified) water in which no interferences are present at or above reporting limits.

11.1.2 All reagent water used to dilute samples and blanks must be pre-extracted with methylene chloride, to minimize interference from phthalate ester contamination.

11.1.2.1 To a 1000 mL separatory funnel, add 1000 mL of reagent water.

11.1.2.2 Add 60 mL of methylene chloride and extract by vigorously shaking with frequent venting.

11.1.2.3 Let the methylene chloride settle for at least 10 minutes then drain and discard.

11.1.2.4 The remaining mixture is then ready for diluting samples and for use as blanks.

11.2 Methylene chloride: pesticide grade or equivalent

11.3 Sodium sulfate: ASC grade, anhydrous granular, rinsed

11.3.1 Sodium sulfate must be solvent-rinsed before use. Assemble the rinsing apparatus and clamp securely.

11.3.2 Insert filter paper in the Buchner funnel and add the entire contents of a 2.5 Kg sodium sulfate container to the funnel.

11.3.3 Add 1 L of methylene chloride to the container, rinsing the insides down with methylene chloride from a PTFE squirt bottle.

11.3.4 Pour the methylene chloride in the container over the sodium sulfate in the Buchner funnel, letting it drain without applying vacuum. Add more methylene chloride, if necessary to completely immerse the sodium sulfate.

11.3.5 After most of the solvent has drained, apply vacuum and rinse with an additional 100 mL from a PTFE squirt bottle. Maintain vacuum until the solvent stops draining.

11.3.6 Transfer the rinsed sodium sulfate to a drying pan and heat in a drying oven at 120° C for at least one hour.

Note: The oven must be vented to the outside to prevent methylene chloride fumes from entering the laboratory.

11.3.7 Remove from the oven with heat-resistant gloves and cool in a hood. Always place a caution sign by the pan while cooling.

11.3.8 After cooling, return to the original container using a clean glass funnel.

11.3.9 Label the container "Rinsed" with date and analyst's initials then place in reagent storage.

11.4 Sodium hydroxide solution, 10N: (prepared from ACS grade pellets)

11.4.1 Carefully add 400 g sodium hydroxide to 1 L of laboratory reagent water that has not been methylene chloride extracted.

11.4.2 Stir on a magnetic stir plate until dissolved then store in a closed and labeled glass container.

Caution: Adding sodium hydroxide to water will generate significant heat. Add pellets slowly and cool to room temperature before using.

11.5 Sulfuric acid solution, 1:1 (v/v): Reagent ACS

11.5.1 Slowly add 500 mL of concentrated sulfuric acid to 500 mL of laboratory reagent water that has not been extracted with methylene chloride.

11.5.2 Stir carefully when cool then store in a closed, labeled glass container.

Caution: Adding sulfuric acid will generate significant heat. Add slowly to avoid splattering and cool before using. **ALWAYS ADD SULFURIC ACID TO WATER (NEVER ADD WATER TO SULFURIC ACID).**

11.6 Methanol: pesticide grade or equivalent

12.0 STANDARDS PREPARATION

12.1 Laboratory-made surrogates and spike solutions are made by the following rules:

12.1.1 Obtain glassware and materials required for surrogates or spike solutions being made.

12.1.2 Use the appropriate solvent for dilution.

12.1.3 Label all glassware and vials holding surrogate/spiking solutions.

12.1.4 Use minimal headspace in vials.

12.2 Surrogate and spiking solutions are stored in 40 mL narrow-mouth amber vials, labeled with the following information:

12.2.1 Surrogate or spike name

12.2.2 Laboratory-assigned surrogate or spike ID

12.2.3 Date prepared

12.2.4 Analyst initials

12.2.5 Solvent used

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 Surrogate/spike data are recorded in an extraction standards logbook. Solutions are labeled with a two-letter prefix followed by a number depending upon logbook page and line. Surrogate/spiking solutions are stored in the prep lab refrigerator at 4° ±2° C. A dilution of spiking solution must be analyzed by the GC/MS laboratory to check concentration before use by the extraction lab. Concentrations must be within established laboratory acceptance limits of 80-120%.

12.4 Neat compounds must be ACS grade or better and be weighed with the calibrated analytical balance in the hood. If neat compound purity is below 95%, concentration must be accounted for in analytical calculations. Locate neat chemicals within the laboratory by use of the chemical inventory list. Do not use any neat compound past its expiration date. If using the last of a compound, see that it is promptly re-ordered.

12.4.1 For neat solids, weigh to the nearest 0.0001 g in a volumetric flask and record the mass. Swirl the flask to mix after adding solvent. If a compound does not dissolve, place the flask in a small sonicator bath to aid in dissolution. Closely monitor the solution and remove as soon as the compound has dissolved. Over-sonicating will generate excessive heat. Dilute to volume and mix well by inverting at least three times. Discard the volume in the neck of the flask and transfer to pre-labeled vials. Store the vials at 4° ±2° C.

12.4.2 For neat liquids, weigh into a tared volumetric flask half-full of appropriate solvent. Add drop-wise using a Pasteur pipette directly into the solvent, being careful not to touch the sides of the flask. Record the mass, swirl then dilute to volume. Mix well and discard the volume in the neck of the flask. Transfer to pre-labeled vials and store at 4° ±2° C.

12.5 Shelf-life of surrogate and spiking solutions is six months for working solutions and one year for stock concentrations. Solutions will be disposed of sooner however, if a manufacturer's expiration date occurs within that time for neat materials used. Laboratory-assigned expiration dates cannot exceed the manufacturer's expiration date. Once a chemical or solution has expired, it must be removed from the laboratory and disposed of. Monitor expiration dates of all chemicals and solutions. To avoid costly shipping, it is important to promptly order more chemical once consumed.

12.6 Often when making surrogate and spiking solutions, serial dilutions are required to achieve proper concentration. Serial dilutions are made from a higher concentration. The following example illustrates making a 1.0 mg/L working solution from a 10,000 mg/L stock concentration.

$$10000\ \text{mg/L} \times \frac{1\ \text{mL stock standard}}{100\ \text{mL final volume}} = 100\ \text{mg/L}$$

$$100\ mg/L \times \frac{1\ mL\ stock\ standard}{100\ mL\ final\ volume} = 1.0\ mg/L$$

12.7 When making a dilution, all data must be entered into Element™.

12.7.1 If a dilution of a dilution is made then the resultant solution would be given another number.

12.7.2 For solution preparation, use only pesticide-grade or better solvents.

12.7.3 Before using a solution, calculations performed when calculating concentration must be verified by a second analyst. Once calculations have been verified, solution concentration must be verified by actual analysis. If analytical recovery is not within laboratory established acceptance limits, a solution cannot be used. Acceptance limits are based on 80% to 120% of calculated values.

12.8 At the end of a shift, vials containing 5 mL or less must be disposed of to minimize contamination and concentration in the solution.

12.9 Acid and base compound surrogate solutions are made separately, from neat materials.

12.9.1 Add 0.5 g of each base-neutral surrogate compound to a 250 mL volumetric flask half-filled with methanol. Swirl, shake or sonicate to dissolve then dilute to volume with methanol.

12.9.2 Add 1.0 g of each acid surrogate compound to another 250 mL volumetric flask half-filled with methanol. Swirl, shake or sonicate to dissolve then dilute to volume with methanol.

12.9.3 Add 100 mL of each solution to a 2 L volumetric half-filled with methanol. Swirl to mix then dilute to volume with methanol. Have this solution checked by GC/MS. Once approved, the prep lab and the mass spec lab must coordinate to be sure the same surrogate mixture is used for calibration and spiking.

12.9.4 Transfer the entire 2 L solution to 40 mL amber vials with PTFE-lined screw-cap lids and store in the prep laboratory refrigerator.

Surrogate	**Mass (g)**	**Dilution Volume (mL)**	**Stock Concentration (ug/mL)**	**Secondary Dilution (mL:mL)**	**Final working Concentration (ug/mL)**
Decafluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
o-terphenyl	0.5	250 in methanol	2000	100:2000	100
Nitrobenzene-d_5	0.5	250 in methanol	2000	100:2000	100
2-Fluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
2,4,6-Tribromophenol	1.0	250 in methanol	4000	100:2000	200
Phenol-d_6	1.0	250 in methanol	4000	100:2000	200
2-Fluorophenol	1.0	250 in methanol	4000	100:2000	200

12.10 Spike solutions with reference to EPA Method 8270C are purchased in individual base/neutral and acid ampules except for naphthalene, which is diluted in methylene chloride from the neat compound.

12.10.1 Weigh 1.0 g of naphthalene into a 100 mL volumetric flask. Add approximately half-volume of methylene chloride to the flask and swirl, shake or sonicate to dissolve then dilute to volume with methylene chloride. Concentration of this solution is 10,000 ug/mL naphthalene.

12.10.2 Pipette 1.0 mL of acid ampule #1 into a 50 mL volumetric flask half-filled with methanol.

12.10.3 Into the same 50 mL volumetric flask, pipette 1.0 mL of base/neutral ampule #2.

12.10.4 Pipette 0.5 mL of the naphthalene solution into the flask then dilute to volume with methanol.

12.10.5 Working spike solutions are checked by GC/MS before using. If all spike compounds are within 80 to 120% of calculated values then the solution is approved for use.

12.10.6 Transfer the entire solution to 40 mL amber vials with PTFE-lined screw-cap lids and store in the prep.laboratory refrigerator.

Compound	**Mass**	**Dilution Volume (mL)**	**Stock/Ampule Concentration (ug/mL)**	**Secondary Dilution (mL:mL)**	**Final working Concentration (ug/mL)**
Naphthalene	1.0 g	100($MeCl_2$)	10000	2.0:200	100
4-Chloro-3-methylphenol (#1)			7500	5.3:200	200
2-Chlorophenol (#1)			7500	5.3:200	200
4-Nitrophenol (#1)			7500	5.3:200	200
Pentachlorophenol (#1)			7500	5.3:200	200
Phenol (#1)			7500	5.3:200	200
Acenapthene (#2)		5000		4.0:200	100
1,4-Dichlorobenzene (#2)			5000	4.0:200	100
2,4-Dinitrotoluene (#2)			5000	4.0:200	100
N-Nitrosodi-n-propylamine (#2)			5000	4.0:200	100
Pyrene (#2)			5000	4.0:200	100
1,2,4-Trichlorobenzene (#2)			5000	4.0:200	100

12.11 Spike solutions with reference to EPA Method 625 are purchased from NSI (Catalog # C-397) at 100 ug/mL. These solutions are used as received and expiration is 6 months from date received. No dilution is required.

12.12 Spike solution with compounds for reference to SW-846 Method 1311 is purchased from Ultra Scientific (Catalog # TCLP-512).

12.12.1 Pipette 1250 µL of 2000 ug/mL purchased solution into a 25 mL volumetric flask half-filled with methanol and dilute to volume.

12.12.2 Working spike solutions are checked by GC/MS before using. If all spike compounds are within 80 to 120% of calculated values then the solution is approved for use.

12.12.3 Transfer the entire solution to a 40 mL amber vial with PTFE-lined screw-cap lids and store in the prep laboratory refrigerator.

Compound	Ampule Concentration (mg/L)	Secondary Dilution (mL:mL)	Final working Concentration (mg/L)
o-cresol	2000	1.25:25	100
m-cresol	2000	1.25:25	100
p-cresol	2000	1.25:25	100
1,4-dichlorobenzene	2000	1.25:25	100
2,4-dinitrotoluene	2000	1.25:25	100
hexachlorobenzene	2000	1.25:25	100
hexachlorobutadiene	2000	1.25:25	100
hexachloroethane	2000	1.25:25	100
nitrobenzene	2000	1.25:25	100
pentachlorophenol	2000	1.25:25	100
pyridine	2000	1.25:25	100
2,4,5-trichlorophenol	2000	1.25:25	100
2,4,6-trichlorophenol	2000	1.25:25	100

12.13 Additional spiking solutions for client-specific analytes will be prepared as needed.

13.0 ANALYTICAL PROCEDURE

13.1 Rinse all glassware with methylene chloride before use.

13.2 Label each Erlenmeyer flask with the following information:

13.2.1 Sample number

13.2.2 "S" for surrogate

13.2.3 "A" for acid when appropriate

13.2.4 "B" for base when appropriate

13.2.5 "L" for spike when appropriate

13.2.6 Method

13.2.7 Final volume

13.3 Label Kuderna-Danish (K-D) flasks and concentrator tubes:

13.3.1 Sample number

13.3.2 Parameter (BNA, TCLP, as appropriate)

13.3.3 Final volume

13.4 Determine sample volume as outlined below.

13.4.1 Shake all sample jars well. If a shaken sample remains clear, mark the water level on the outside of the jar then transfer its entire contents to a separatory funnel. Do not discard the sample jar. After extraction is complete, fill the jar with tap water to the water level mark then measure with a graduated cylinder to attain the sample volume.

13.4.2 If a shaken sample is so cloudy it is difficult to see through the jar, let the sediment settle for approximately five minutes then decant the aqueous portion into a clean graduated cylinder to measure the volume. Shake any sediment remaining in the jar into the trash. DO NOT RINSE A SAMPLE JAR TO REMOVE SEDIMENT OR RESIDUE. After sample volume has been measured and recorded, transfer the graduated cylinder contents to a separatory funnel.

13.4.3 If less than 500 mL of a sample is measured, consult the project chemist before extracting. Extracting less than 500 mL will result in elevated reporting limits, which must be client approved.

Note: TCLP leachates extracted using 500 mL do not require consultation with the project chemist before extracting.

13.5 Add 100 µL of surrogate solution to each separatory funnel with a 100 µL syringe, to extracts taken to 1.0 mL final volume. Add 1.0 mL of surrogate to each separatory funnel with a 1000 µL syringe, to extracts taken to 10.0 mL final volume.

13.5.1 Syringes used must be dedicated to BNA surrogate additions.

13.5.2 Surrogate is added to all extractions including samples, Extraction Blanks (BLK), Blank Spikes (BS) and Matrix Spikes.

13.5.3 Do not submerge the syringe into a liquid when injecting surrogate. Slowly inject approximately one inch above the liquid's surface.

13.5.4 Do not inject too fast since splashing may occur. If splashing occurs onto the syringe, rinse it internally and externally with methanol. Discard the rinsate.

13.5.5 Cross out the "S" on each corresponding Erlenmeyer flask as surrogate is added.

13.6 Add 100 µL of spiking solution to Matrix Spikes, Matrix Spike Duplicates and Blank Spikes, if final volume will be 1.0 mL. Add 1.0 mL of spiking solution to Matrix Spikes, Matrix Spike Duplicates and Blank Spikes, if final volume will be 10.0 mL. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.7 Acidify samples by adding approximately 2 mL of 1:1 sulfuric acid solution to each separatory funnel using a Pasteur pipette. Mix well by swirling and/or shaking.

13.8 Rinse the sample jar and/or graduated cylinder with methylene chloride:

13.8.1 Add approximately 100 mL methylene chloride to each sample jar. Replace the lid (very important).

13.8.2 Shake and vent as necessary. If a graduated cylinder was used to measure the sample volume, transfer the methylene chloride from the sample container into the graduated cylinder. Swirl the methylene chloride to rinse the cylinder sides.

13.8.3 Transfer the methylene chloride into the separatory funnel containing sample.

13.9 Acid Extraction:

13.9.1 Stopper the separatory funnel and invert once. Immediately open the stopcock to release solvent pressure.

13.9.2 Close the stopcock, shake and vent again.

13.9.3 Repeat until there is no evidence of pressure.

13.9.4 Shake for one to two minutes.

13.9.5 Let the separatory funnel sit for at least ten minutes for phase separation.

13.9.5.1 Check the pH of each solution using a pH indicator strip by removing a few drops from the separatory funnel with another clean Pasteur pipette. Do not dip the pH strip directly into a separatory funnel to take a reading.

13.9.5.2 The pH should be less than 2. If not, add additional 2 mL aliquots of acid until a pH of less than 2 is attained then cross out the "A" on the corresponding Erlenmeyer flask. Add no more acid than one percent of the initial sample volume (10 mL for a 1000 mL initial volume) as larger additions constitute a significant extraction volume change.

13.9.5.3 Consult with the project chemist if a sample cannot be adjusted to a pH less than 2 with the maximum acid volume.

13.9.6 If a heavy emulsion has formed, drain the emulsion into a 125 mL separatory funnel and shake vigorously. Let sit for at least ten minutes.

13.9.6.1 If separation occurs in the 125 mL funnel, drain the solvent phase into the Erlenmeyer flask. Drain the water back into the 2 L funnel.

13.9.6.2 If separation does not occur, drain the entire volume from the 125 mL funnel into a PTFE centrifuge tube (this may require multiple tubes). Centrifuge for five minutes at 4500 rpm. Once centrifuged, transfer the water phase with any intermediary emulsion back into the 2 L funnel. Pour the bottom phase into the Erlenmeyer flask.

13.9.7 If a light emulsion has formed, drain the methylene chloride (bottom layer) into the corresponding 500 mL Erlenmeyer flask, passing it through phase separation paper. Rinse the paper thoroughly.

13.9.8 If there is no emulsion, drain the methylene chloride directly into the Erlenmeyer flask.

13.10 Repeat the extraction twice more, each time using approximately 60 mL of methylene chloride.

13.10.1 Repeat the process of rinsing the sample container and (if used) graduated cylinder with methylene chloride, then transfer to the separatory funnel for the second extraction.

13.10.2 Add the methylene chloride directly to the separatory funnel for the third extraction.

13.10.3 Approximately 200 mL of acid solvent extract will be collected.

13.11 After the acid extraction is complete, adjust pH to greater than 11 by adding approximately 7 mL of 10N sodium hydroxide solution to each separatory funnel using a graduated cylinder. Mix well by swirling and/or shaking.

13.12 Base-Neutral Extraction

13.12.1 Add 60 mL of methylene chloride.

13.12.2 Stopper the separatory funnel and invert. Immediately open the stopcock to release pressure.

13.12.3 Close the stopcock, shake and vent again.

13.12.4 Repeat until there is no evidence of pressure venting.

13.12.5 Shake for an additional two minutes.

13.12.6 Let the separatory funnel sit for at least ten minutes, for phase separation.

13.12.6.1 Check the pH of each solution using a pH indicator strip by removing a few drops from the separatory funnel with another clean Pasteur pipette. Do not dip the pH strip directly into separatory funnel to take a reading.

13.12.6.2 The pH should be greater than 11. If not, add additional 1 mL aliquots of sodium hydroxide until a pH of greater than 11 is attained, then cross out the "B" on the corresponding Erlenmeyer flask. Add no more than one percent of the initial extraction volume (10 mL for a 1000 mL initial volume) as larger additions constitute sample dilution.

13.12.6.3 Consult with the project chemist if a sample cannot be adjusted to a pH greater than 11 with the maximum base volume.

13.12.7 If a heavy emulsion forms during extraction, drain the emulsion into a 125 mL separatory funnel and shake vigorously. Let sit for at least ten minutes.

13.12.7.1 If separation occurs in the 125 mL funnel, drain the solvent phase into the Erlenmeyer flask. Drain the water back into the 2 L funnel.

13.12.7.2 If separation does not occur, drain the entire contents of the 125 mL funnel into a PTFE centrifuge tube. Centrifuge for five minutes at 4500 rpm. Once centrifuged, transfer the water phase with any intermediary emulsion back into the 2 L funnel. Pour the bottom phase into the Erlenmeyer flask.

13.12.8 If a light emulsion has formed, drain the methylene chloride (bottom layer) into the 500 mL Erlenmeyer flask containing acid compound extract, passing it through phase separation paper. Rinse the paper thoroughly.

13.12.9 If there is no emulsion, drain the methylene chloride directly into the Erlenmeyer flask.

13.13 Repeat the base-neutral extraction twice more, each time using approximately 60 mL of methylene chloride. A total of approximately 400 mL of BNA extract will be collected for concentration.

13.14 When extractions are complete, if a graduated cylinder was not used to determine sample volume, determine the volume by filling the sample container with tap water up to the water level mark. Transfer to a graduated cylinder and record the volume.

13.15 Remove all sample identification tags from the empty sample jar and discard both.

13.16 Transfer extract to a K-D concentrator:

13.16.1 Quantitatively transfer each extract from the 500 mL flasks through a funnel containing about one inch of sodium sulfate in P8 filter paper into Kuderna-Danish concentrators, with an attached 10 mL graduated concentrator tube.

13.16.2 Rinse the flask and funnel with several 20-30 mL aliquots of methylene chloride to complete the transfer.

13.17 Concentrate the extracts

13.17.1 Add one or two clean boiling chips to each concentrator tube and attach a three-ball Snyder column.

13.17.2 Pre-wet the Snyder columns by adding approximately 1 mL of methylene chloride through the top.

13.17.3 Place the concentrators in a water bath (70-80° C) where the concentrator tube is partially immersed and the entire lower rounded surface of the K-D flask is bathed with vapor.

13.17.4 At the proper rate of distillation, the column balls will actively chatter but not flood. Until chattering begins, rattle the Snyder columns periodically.

13.17.5 When the apparent volume of liquid reaches 3-4 mL (in ten to fifteen minutes), remove the K-D and let drain for at least 10 minutes.

13.17.6 Remove the Snyder column, rinsing the lower joint into the concentrator tube with 2 to 3 mL of methylene chloride.

13.18 Micro-Snyder column technique is used to adjust final extract volumes to 1.0 mL. Blowdown is not used when adjusting final volume to 1.0 mL, to minimize analyte loss from concentration.

13.18.1 Micro-Snyder Column Technique:

13.18.1.1 Add another clean boiling chip then attach a 2-ball micro-Snyder column.

13.18.1.2 Pre-wet the column with 0.5 mL methylene chloride.

13.18.1.3 Place concentrator tubes in a water bath, partially immersed. Water temperature should be 60-70° F. Care should be taken to avoid bumping or column flooding due to immersing too deep.

13.18.1.4 When the apparent volume reaches 0.5 mL, remove from the bath. Cool for at least ten minutes.

13.18.1.5 Rinse the ground-glass joints with 0.2 mL methylene chloride after removing Snyder columns.

13.18.1.6 Transfer extracts to a 1.0 mL calibrated vial using a clean disposable Pasteur pipette.

13.18.1.7 Using approximately 0.2 mL of methylene chloride, rinse each concentrator tube, then transfer to the vial.

13.18.1.8 Adjust the final volume to 1.0 mL with methylene chloride then cap tightly.

13.18.1.9 Extracts are ready for analysis and must be stored in the GC/MS sample refrigerator at 4 ±2° C.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Extraction analysts are responsible for extraction documentation and data integrity. All documentation must be correctly filled in. It is important to document extractions by correctly filling in, turning in and filing accurate paperwork. This is mandatory for quality control and to provide clients with defensible data.

14.2 Report extractions as follows:

14.2.1 Input all extraction data to Element™. Extraction reports must be filled in completely to insure that results are reported correctly and data is associated with the right quality control batch number.

14.2.2 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in completely and correctly.

14.3 Complete all laboratory records as follows:

14.3.1 Logbooks must be filled in completely and correctly. Corrections are to be made with a line-out, not a write-over or scribble-out. Blank lines in the logbook must be Z'd out.

14.3.2 Transfer extraction summary benchsheets (including CoC forms) to the GC/MS analyst with finished extracts.

15.0 QUALITY ASSURANCE

15.1 Extraction analysts must extract an Extraction Blank and Blank Spike daily, once per shift or every 20 samples (whichever is more frequent) to demonstrate that extraction interferences are under control.

15.2 Matrix Spikes and Matrix Spike Duplicates (provided enough sample volume is received) are extracted at a minimum of once every twenty samples or once a week, depending upon the frequency of sample receipt.

15.2.1 Performance records are maintained to document data quality generated.

15.2.2 Matrix spiked samples are prepared for analysis by first adding spike solution and surrogate before extraction.

15.3 Blank Spikes are prepared by extracting the addition of BNA spike solution and surrogate solution to 1 L of pre-extracted laboratory reagent water.

15.4 Matrix interference from samples or from laboratory-induced contamination may affect analyte recovery. Investigation and re-extraction may be necessary if surrogate or spiked compounds fail to pass laboratory established acceptance limits.

15.5 Surrogate must be added to all extractions.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before preparation of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by extracting a demonstration of capability study.

16.2 Prepare a spiking solution that will give final extract concentrations, mid-range to the GC/MS calibration. This solution must be prepared independent of GC/MS calibration standards. Prepare four (4) 1000 mL aliquots of pre-extracted water. Inject 100 µL of spiking solution into each. Add surrogate, then extract as samples, going through all extraction steps in this procedure, to a final volume of 1.0 mL.

16.3 After extraction, have the four (4) extracts analyzed for all spiked compounds in accordance with all GC/MS procedure requirements.

16.4 Input results to the IDC spreadsheet to calculate average percent recovery (x) and percent relative standard deviation of recovery (s), for each analyte, using the four results.

16.4.1 For each analyte, (x) must be within historical statistical recovery limits and (s) must be less than or equal to 20%. If (s) and (x) for all analytes meet acceptance, the demonstration of capability study is acceptable. The analyst is authorized to extract samples by the procedure.

16.4.2 If one or more analytes fail at least one acceptance limit, the analyst must proceed through the following steps.

16.4.2.1 Locate and correct any source of error then repeat the demonstration of capability only for analytes not meeting acceptance. If the failed analytes are acceptable on the second attempt, the study is complete and acceptable.

16.4.2.2 Repeated failure will indicate general problems with the extraction procedure or analyst technique. If this occurs, correct and revise any faulty steps in the procedure or technique.

16.4.2.3 Once corrective action has been taken, re-extract the demonstration of capability study for all analytes. If all analytes are acceptable, the study is complete and acceptable.

16.5 Samples may not be prepared by any analyst until an acceptable demonstration of capability study has been successfully completed.

16.6 A successful continuing demonstration of capability (CDC) study must be completed annually by all analysts by one of the following approaches:

16.6.1 By repeating the IDC study.

16.6.2 By using four consecutive blank spike results obtained during the course of routine sample extraction and when extracted exclusively by the analyst.

16.6.3 By extracting an acceptable blind performance testing sample.

16.6.4 By exclusively extracting a method detection limit (MDL) study. The last four results of the study may be used.

16.7 A Method Detection Limit (MDL) study is required annually in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor chemical use.

17.2 Reference any specific written disposal instructions before disposing of laboratory chemicals.

17.3 Conserve the use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material Safety Data Sheets are located on the laboratory intranet library.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount required.

18.3 Follow all instructions in TriMatrix Laboratory SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition.* Final Update III, Revision 3, December 1996, Method 3510C, "Separatory Funnel Liquid-Liquid Extraction"

19.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids

20.0 ATTACHMENTS

20.1 Preparation Batch Report Example

20.2 Standards Log Report Example

20.3 Water Extraction Logbook Example

Attachment 20.1
Preparation Batch Report Example

TriMatrix Laboratories, Inc. **PREPARATION BATCH** 0801114 Page 1 of 1 Printed: 11/7/2008 10:14:54AM

Semivolatiles MS, Waste Water, 625 Liquid/Liquid Extraction

Batch comments:
(none)

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	Client Source ID	Client Spike ID	Client nL Spike	Comments	
0801114-BLK1		Feb-01-08 08:23	KB9	1000	5					
0801114-BLK2		Feb-05-08 08:23	ASC	1000	1				same as batch 0801235	
0801114-BS1		Feb-01-08 08:23	KB9	1000	5		8010650	500		
0801114-BS2		Feb-05-08 08:23	ASC	1000	1		8010650	100	same as batch 0801235	
0801114-BSD1		Feb-01-08 08:23	KB9	1000	5		8010650	500		
0801114-DUP1		Feb-01-08 08:23	KB9	1000	5	0801501-19				
0801114-DUP2		Feb-01-08 08:23	KB9	1000	5	0801501-20				
0801114-DUP3		Feb-01-08 08:23	KB9	1000	5	0801501-25				
0801114-DUP4		Feb-01-08 08:23	KB9	1000	5	0801501-26				
0801114-DUP5		Feb-05-08 08:23	ASC	1000	1	0801501-27				
0801501-19	A	Feb-01-08 08:23	KB9	1000	5	[redacted]			Added for BatchQC in: 0801114	BatchQC
625 SVOCs (master list)		*625 SVOCs (TTOs)*		*625 SVOCs (TTOs) custon*						
0801501-20	A	Feb-01-08 08:23	KB9	1000	5	[redacted]			Added for BatchQC in: 0801114	BatchQC
625 SVOCs (master list)		*625 SVOCs (TTOs)*		*625 SVOCs (TTOs) custon*						
0801501-25	A	Feb-01-08 08:23	KB9	1000	5	[redacted]			acids, base neutrals	acids, base neutrals
625 SVOCs (master list)		*625 SVOCs (TTOs)*		*625 SVOCs (TTOs) custon*						
0801501-26	A	Feb-01-08 08:23	KB9	1000	5	[redacted]			acids, base neutrals	acids, base neutrals
625 SVOCs (master list)		*625 SVOCs (TTOs)*		*625 SVOCs (TTOs) custon*						
0801501-27	A	Feb-05-08 08:23	ASC	1000	1	[redacted]			Added for BatchQC in: 0801114	BatchQC
625 SVOCs (master list)		*625 SVOCs (TTOs)*		*625 SVOCs (TTOs) custon*						

Comments:	Analyst Initials:

bch_TM_byAnalysis.rpt

Approved By: [signature] 2-12-09 **QA Officer**

Approved By: BJL+ 2/12/09 **Area Supervisor**

Attachment 20.2
Standards Log Report Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

8060471

Description:	BNA Surrogate	Expires:	Nov-10-08
Standard Type:	Surrogate Spike	Prepared:	Jun-16-08
Solvent:	MeOH Lot#076070	Prepared By:	Andrea S. Colborn
Final Volume (mls):	2000	Department:	Semivolatiles MS
Vials:	1	Last Edit:	Jun-16-08 17:19 by ASC

Analyte	CAS Number	Concentration	Units
Phenol-d6	13127-88-3	200	ug/mL
o-Terphenyl	84-15-1	100	ug/mL
Nitrobenzene-d5	4165-60-0	104	ug/mL
Decafluorobiphenyl	434-90-2	100	ug/mL
2-Fluorophenol	367-12-4	202	ug/mL
2-Fluorobiphenyl	321-60-8	100	ug/mL
2,4,6-Tribromophenol	118-79-6	201	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
7120351	Base Surrogate Stock	Dec-14-07	Brian J. Hall	Dec-14-08	Dec-14-07 11:52 by ASC	100
7120371	Acid Surrogate Stock	Dec-18-07	Andrea S. Colborn	Nov-10-08	Dec-18-07 08:40 by ASC	100

Approved By: _____ 2-12-09
QA Officer

Approved By: _____ 2/12/09
Area Supervisor

Attachment 20.3
Water Extraction Logbook Example

TriMatrix Laboratories, Inc.

Organic Extraction Lab
Water Extraction Logbook Date: 12/29/03

Client	Project/ Submittal	Sample Number Range	LIMS Batch ID	Test or Parameter	Surrogate Spike Number and Volume		Matrix Spike Number and Volume	
RMT	36466-13	352226 - 233	211038	8270BNA 3510	PL4.2-7	100mL	PL3.12-9	100mL
Rose Twp	29928-597	352327 - 328	"	8270BN 3510	"	"		
RMT	36466-13	352228 - 230	210615	8015 DRO 3550			GC 2.41-16	500mL

12/29/03

LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials
352226	82	990	1.0mL	MBS
352227		1040		
352228		1040		
352231		1060		
352233		1060		
MPB129		1000		
LFB129		1000		
LFB129	↓	1000	↓	↓
352327	82	1030	1.0mL	MBS
352328	↓	1040	↓	↓
352228	82	1020	1.0mL	MBS
352230		255		
MPB129		1000		
LFB129		1000		
LFB129	↓	1000	↓	↓

DJM 12/29/03

LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials

DJM 12/29/03

Reagent Information

CH_2Cl_2 Lot #:	030986	1:1 H_2SO_4 Reagent #:	PLR1.42-13
Hexane Lot #:	030702	1:1 HCl Reagent #:	NA
Na_2SO_4 Reagent #:	PLR1.42-16	10N NaOH Reagent #:	PLR1.44-11
		NaCl Reagent #:	NA

file: Prep Lab Water Logbook page: 3 of 30 revision: 0.0

Approved By: DD 2-12-09 QA Officer

Approved By: BJH 2/12/09 Area Supervisor

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Extraction of BNA Semi-Volatiles from Soil, Sediment and Sludge

SW-846 Method 3550B

APPROVALS:

Area Supervisor: Brian Hall Date: 2/12/09
Brian J. Hall

QA Officer: Date: 2-12-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 2/12/09
Jeff P. Glaser

Procedure Number: GR-09-103
Revision Number: 3.7

Date Initiated: 1/18/96
Effective Date: 3/12/09

Date Revised: 2/12/09
Pages Revised: All

By: Daniel J. Mierendorf
Total Number of Pages: 19

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the ultrasonic extraction of BNA non-volatile and semi-volatile organic compounds from soils, sediments and sludge by sonication.

1.2 Ultrasonic extraction is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludge and waste. The ultrasonic process ensures intimate contact of the sample matrix with the extraction solvent.

1.3 This procedure is not appropriate where high extraction efficiencies for very low analyte concentrations are necessary.

1.4 Ultrasonic extraction is not as rigorous as other extraction techniques for soils/solids. Therefore, it is critical that the procedure (including the manufacturer's instructions) be followed explicitly to achieve the maximum extraction efficiency. Critical aspects of the extraction are as follows:

1.4.1 The extraction device must have a minimum of 300 watts of power and be equipped with appropriate size disrupter horns

1.4.1.1 Use a 3/4" horn for concentrations less than or equal to 20 mg/kg.

1.4.1.2 Use a 1/8" tapered microtip attached to a 1/2" horn for concentration greater than 20 mg/kg.

1.4.2 The horn must be properly maintained, including tuning according to the manufacturer's instructions prior to use and inspection of the horn tip for excessive wear.

1.4.3 Samples must be properly prepared by thorough mixing with sodium sulfate so it forms a free-flowing powder prior to addition of the solvent.

1.4.4 Extraction horns are not interchangeable. The referenced method indicates that use of the 3/4" horn is inappropriate for the high concentration extraction. Particularly for extraction of very nonpolar organic compounds such as PCBs, which are strongly adsorbed to the soil matrices.

1.4.5 Three extractions are performed with the appropriate solvent, the extraction is performed in the specified pulse mode and the horn tip is positioned just below the surface of the solvent yet above the sample.

1.4.6 Very active mixing of the sample and the solvent must occur when the ultrasonic pulse is activated. The analyst must observe such mixing at some point during the extraction process.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 2, December, 1996, Method 3550B, "Ultrasonic Extraction"

3.0 SUMMARY OF PROCEDURE

3.1 The extraction for known analyte concentrations at or below 20 mg/kg is as follows:

3.1.1 Anhydrous sodium sulfate is mixed with 30 g of sample to form a free-flowing powder.

3.1.2 The powder is serially extracted three times by ultrasonic extraction with 1:1 methylene chloride/acetone and concentrated to 10 mL for gas chromatrography/mass spectrometry (GC/MS) analysis.

3.1.3 This technique is applicable to low to medium analyte concentrations and minimal matrix interference. If necessary, the final extract volume may be greater than 10 mL.

3.1.4 If the minimum reporting limit is required, the final extract may be concentrated to a final volume of 1.0 mL.

3.2 The extraction for known analyte concentrations above 20 mg/kg or for difficult sample matrices is as follows:

3.2.1 Samples with a history of high analyte concentration or matrix interference are extracted using 1-2 g of sample. Analyst discretion based on experience may be used to determine the need for this extraction.

3.2.2 Anhydrous sodium sulfate is mixed with the sample to form a free-flowing powder.

3.2.3 The powder is extracted once by ultrasonic extraction with 1:1 methylene chloride/acetone and and concentrated to 10 mL for gas chromatrography/mass spectrometry (GC/MS) analysis. If necessary, the final extract volume may be greater than 10 mL.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to SOP GR-04-103, Attachments 23.1 and 23.2

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-04-103, *Semi-Volatile Laboratory Ion Trap Mass Spectrometry Analysis of Base/Neutral/Acid Compounds*, latest revision

5.2 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration,* latest revision

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest edition

5.4 TriMatrix SOP GR-16-100, Gl*assware Cleaning and Preparation for the Organics Extraction Laboratory*, latest edition

5.5 TriMatrix SOP GR-09-128, *Mixing and Grinding Samples for Organic Extractions,* latest edition

5.6 TriMatrix SOP GR-04-101, *Semi-Volatiles Laboratory Corrective Actions*, latest revision

5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interferences can be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware, leading to discrete artifacts and/or elevated baselines in the total ion current profiles.

6.2 Do not use plastic materials, including polypropylene squirt bottles. Phthalate esters leaching from plastics can result in false positives.

6.3 All materials used in extraction and analysis must routinely be demonstrated to be interference-free by running method performance blanks.

6.4 Rinse all washed glassware with pesticide grade or better methylene chloride immediately before use.

6.5 Use only high purity reagents (reagent grate or better) and solvents (pesticide grade or better). Solvents used for extraction and analysis must be tested prior to use. Once proven to be contaminant-free, the solvent lot is sequestered from the vendor to ensure consistent purity.

6.6 Clean glassware in accordance with TriMatrix SOP GR-16-100.

6.7 The severity of matrix interference will vary considerably from sample to sample. Re-extraction may be necessary if surrogates or other spiked compounds fail established laboratory recovery limits

7.0 SAFETY PRECAUTIONS

7.1 Laboratory coats and approved safety glasses must be worn in the extractions laboratory. Disposable gloves must be worn whenever samples, solvents or reagents are handled.

7.2 Follow all safety instructions outlined in the TriMatrix laboratory safety manual and chemical hygiene plan.

7.3 The toxicity and/or carcinogenicity of reagents used in this procedure has not been precisely defined.

7.3.1 Treat all chemicals as a potential health hazard.

7.3.2 Reduce exposure to the lowest possible level by adherence to all established safety practices.

7.3.3 Material Safety Data Sheets (MSDS) of all chemicals used in this procedure are located on the laboratory intranet library.

7.4 Bring any safety issues to the attention of the area supervisor and/or the health and safety officer.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 To achieve minimum reporting limits, a minimum 30 g of soil sample must be collected and extracted. Additional sample volume is necessary if matrix spikes are required.

8.2 Collect samples in wide-mouth glass jars, with PTFE-lined screw-cap lids. These jars must meet or exceed current USEPA cleanliness standards. Plastic containers and/or plastic-lined lids must not be used due to potential phthalate ester contamination.

8.3 Store samples away from light and at 4 ±2° C. Store extracts away from light and at less than -10° C.

8.4 Analysis must be performed within 40 days of sample extraction.

8.5 Extract samples within fourteen days of the collection date.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Beaker, thick-walled Pyrex: 400 mL

9.2 Ultrasonic disrupter, Fisher model 550 with ¾ inch horn

9.2.1 For the high concentration technique, 1/8 inch tapered microtip attached to a ½ inch horn

9.2.2 Sonabox

9.3 Kuderna-Danish (K-D) concentrator glassware

9.3.1 Concentrator tubes: 10 mL, graduated (Kontes K-570050-1025 or equivalent)

9.3.2 Concentrator flasks: 500 mL (Kontes K-570001-500 or equivalent)

9.3.3 Concentrator tube clips

9.3.4 Snyder columns: Three-ball macro (Kontes K-50300-0121 or equivalent)

9.3.5 Snyder columns: Two-ball micro

9.4 Water bath, capable of variable temperature control to ±5° C.

9.5 Balance, top-loading, capable of accurately weighing to the nearest 0.01 g

9.6 Filter paper: 24 mm, Fisher Brand P8 or equivalent

9.7 Wood spatulas (tongue depressors), disposable

9.8 Pasteur pipets, disposable, 1 mL

9.9 Boiling chips, PTFE, rinsed with methylene chloride

9.10 Vials, with PTFE-lined screw-cap lids, calibrated to 10.0 mL

9.11 Autosampler vials, with PTFE-lined screw-cap septum lids, 2 mL, calibrated to 1.0 mL

9.12 Microsyringes, 100 µL and 1000 µL

9.13 Filtration apparatus

9.13.1 Filter flask: 500 mL side-arm

9.13.2 Buchner funnel, 90 mm

9.13.3 Stopper, PTFE, holed

9.13.4 Filter paper, 0.45 µm for clay samples or samples with fine particulates

9.14 Filter funnel, 100 mm, ribbed

9.15 Sodium sulfate rinsing equipment:

9.15.1 Buchner funnel: 20 cm

9.15.2 Flask: 4 L Vacuum

9.15.3 Drying pan: 13 x 9 inch, Pyrex glass

9.15.4 Vacuum pump

9.15.5 Drying oven

9.15.6 Stopper: Teflon with hole for the Buchner funnel

9.15.7 Clamps: supporting

9.15.8 Filter paper: qualitative fast, 20 cm

9.15.9 Squirt bottle, PTFE

9.15.10 Funnel, glass: rinsed with methylene chloride

9.16 Hot Plate: Capable of variable temperature control to ±5° C

9.17 Volumetric Flasks: 10 mL, 50 mL, 100 mL, 1 L

9.18 Amber vials, with PTFE-lined screw-cap lids, 40 mL

9.19 Analytical balances, capable of accurate measurement to the nearest 0.0001 g

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Ultrasonic disrupters must be tuned at the beginning of each shift.

10.2 Ultrasonic probes must be cleaned with solvent before extracting each new sample and thoroughly cleaned at the end of each shift.

11.0 CHEMICALS AND REAGENTS

11.1 Acetone: Pesticide grade or better

11.2 Methylene chloride: Pesticide grade or better

11.3 Sodium sulfate: ASC grade, anhydrous granular, rinsed as follows:

11.3.1 Assemble the sodium sulfate rinsing apparatus and clamp securely.

11.3.2 Insert filter paper in the Buchner funnel and add the entire contents of a 2.5 kg sodium sulfate container to the funnel.

11.3.3 Add 1 L of methylene chloride to the sodium sulfate container, rinsing down the inside with methylene chloride from a PTFE squirt bottle.

11.3.4 Pour this methylene chloride over the sodium sulfate in the Buchner funnel, letting drain without applying vacuum. Add more methylene chloride if necessary to completely immerse the sodium sulfate.

11.3.5 After most of the solvent has drained, apply vacuum and rinse with an additional 100 mL from the PTFE squirt bottle. Maintain vacuum until solvent stops draining.

11.3.6 Transfer the rinsed sodium sulfate to a drying pan and heat in a drying oven set at ≥130° C for at least one hour.

Note: The drying oven must be vented.

11.3.7 Remove from the oven with heat-resistant gloves and cool in a hood. Always place a caution sign by the pan while cooling.

11.3.8 After cooling, return to the original container using a clean glass funnel.

11.3.9 Label the container with the assigned reagent identification number then place in reagent storage.

11.4 Methylene chloride/acetone, 1:1 (v/v): make by combining equal volumes of the solvents

12.0 STANDARDS PREPARATION

12.1 Laboratory-made surrogates and spike solutions from neat materials are made by the following rules:

12.1.1 Only use an analytical balance that weighs to 0.0001 g. Be sure to record weighings to the nearest 0.0001 g.

Note: The balance must be in a fume hood or otherwise vented.

12.1.2 Obtain glassware and materials required for surrogates or spike solutions being made.

12.1.3 Use the required solvent for dilutions.

12.1.4 Minimize headspace when filling storage vials

12.2 Store surrogate and spiking solutions in 40 mL narrow-mouth amber vials, labeled with the following information:

12.2.1 Surrogate or spike name

12.2.2 Laboratory-assigned surrogate or spike identification number

12.2.3 Date made

12.2.4 Analyst initials

12.2.5 Solvent

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 Once solid surrogate or spike compounds are weighed into a volumetric flask and the weighing recorded, add enough solvent to dissolve the neat material then dilute to volume and invert several times to mix thoroughly.

Note: If solids do not dissolve, place in a small ultrasonic bath to aid in mixing. Do not leave for an extended time because the bath will generate heat. After the sonication, dilute to volume then transfer to tightly capped pre-labeled vials and store at 4 ±2° C.

12.4 Weigh liquid surrogate or spike compounds into volumetric flasks containing an approximately half-volume of solvent. Use a Pasteur pipet to add compound drop-wise, directly into the solvent. Be careful not to touch the inside of the flask or the solvent surface with the pipet tip. Record the weighing and dilute to volume. Invert enough times to mix thoroughly then transfer to pre-labeled tightly capped vials and store at 4 ±2° C.

12.5 Record surrogate/spike preparation data in the extraction standards log. Label solutions with the identification number. Store in the extractions laboratory refrigerator at 4 ±2° C. A dilution of each pre-extraction spiking solution must be analyzed by GC/MS to check concentration prior to use. Concentration must be within established laboratory acceptance limits.

12.6 Neat compounds must be ACS grade or better. If purity of a neat standard is below 95%, concentration must be accounted for in the analytical calculation. Do not use any neat compound past the expiration date. If using the last of a compound, see that it is promptly re-ordered.

12.7 The shelf-life of surrogate and spiking solutions is six months for working solutions and one year for stock concentrations. Solutions will be disposed of sooner however, if a manufacturer's expiration date occurs within that time for the neat material used. Once a chemical or solution has expired, remove it from the laboratory for disposal. Monitor the expiration date of all chemicals and solutions. To avoid costly shipping, it is important to promptly order more chemical once consumed.

12.8 Often when making surrogate and spiking solutions, serial dilutions are required to achieve proper concentration. Serial dilutions are made from a higher concentration. The following example illustrates making a 1.0 mg/L working solution from a 10,000 mg/L stock concentration:

$$10000\ \text{mg/L} \times \frac{1\ \text{mL stock standard}}{100\ \text{mL final volume}} = 100\ \text{mg/L}$$

$$100\ \text{mg/L} \times \frac{1\ \text{mL stock standard}}{100\ \text{mL final volume}} = 1.0\ \text{mg/L}$$

12.9 When making a dilution, enter all data into the laboratory information management system (Element™).

12.9.1 If a dilution of a dilution is made then the resultant solution needs given a separate identification number.

12.9.2 For solution preparation, use only pesticide-grade or equivalent solvents.

12.10 Before using a solution, the calculation performed when calculating concentration must be verified by a second analyst. Once calculations have been verified, solution concentration needs verified by actual GC/MS analysis. If analytical recovery is not within 80 - 120% of the calculated value, the solution cannot be used.

12.11 Dispose of any pre-extraction spiking solution vial containing 5 mL or less at the end of the shift to minimize contamination and analyte concentration. When not in use, keep spike solution vials in the extractions laboratory refrigerator.

12.12 Surrogates are made separately from neat materials and diluted in methanol to make one 2000 mL working solution. Working solutions are checked by GC/MS before using. If all surrogates in a working solution are within 80 - 120% of the calculated value, the solution is acceptable for use. Remaining individual surrogate stock solutions are used by the GC/MS laboratory to calibration the GC/MS.

Surrogate	Mass	Dilution	Stock	Secondary	Final working

	(g)	Volume (mL)	Concentration (mg/L)	Dilution (mL:mL)	Concentration (mg/L)
Decafluorobiphenyl	0.5000	250	2000	100:2000	100
o-terphenyl	0.5000	250	2000	100:2000	100
Nitrobenzene-d_5	0.5000	250	2000	100:2000	100
2-Fluorobiphenyl	0.5000	250	2000	100:2000	100
2,4,6-Tribromophenol	1.0000	250	4000	100:2000	200
Phenol-d_6	1.0000	250	4000	100:2000	200
2-Fluorophenol	1.0000	250	4000	100:2000	200

12.13 Matrix spike solutions are purchased in individual ampules except for naphthalene, which is diluted in methylene chloride from the neat compound. These stock solutions are diluted into one 200 mL volumetric flask in methanol and brought to final volume with methanol. The working spike solution is then checked by GC/MS before using. If all spike compounds are within 80 - 120% of the calculated value, the solution is acceptable for use.

Surrogate	Mass (g)	Dilution Volume (mL)	Stock Concentration (mg/L)	Secondary Dilution (mL:mL)	Final working Concentration (mg/L)
Naphthalene	1.0000	100($MeCl_2$)	10000	2.0:200	100
4-Chloro-3-methylphenol			7500	5.3:200	200
2-Chlorophenol			7500	5.3:200	200
4-Nitrophenol			7500	5.3:200	200
Pentachlorophenol			7500	5.3:200	200
Phenol			7500	5.3:200	200
Acenapthene			5000	4.0:200	100
1,4-Dichlorobenzene			5000	4.0:200	100
2,4-Dinitrotoluene			5000	4.0:200	100
N-Nitrosodi-n-propylamine			5000	4.0:200	100
Pyrene			5000	4.0:200	100
1,2,4-Trichlorobenzene			5000	4.0:200	100

13.0 ANALYTICAL PROCEDURE

13.1 The Fisher Model 550 ultrasonic disrupter must be tuned at the beginning of each shift before samples are processed. Perform the tune as follows:

13.1.1 Turn the **OUTPUT CONTROL** knob counterclockwise to zero.

13.1.2 Press the **POWER SWITCH** to **ON** (up position). The switch will light up.

13.1.3 When the prompt appears, press **TUNE**. The display will read: [**TUNING - - - PROBE ACTIVE**].

13.1.4 Turn the **OUTPUT CONTROL** knob to setting 10 (5 if using the microtip).

13.1.4.1 Note the position of the bar graph on the display screen. Do NOT exceed 70%.

13.1.4.2 Rotate the **OUTPUT CONTROL** knob clockwise or counterclockwise until a minimum (not maximum) reading is obtained. If a reading of less than 20% cannot be obtained, there is a problem with the horn and it must be repaired before using.

13.1.5 Press the **TUNE** key to display prompts for programmed or continuous operation and set the disrupter for programmed operation.

13.1.6 The disrupter is now tuned. Record all tuning information in the tuning logbook.

13.2 Before removing a subsample from a sample container, homogenize the entire sample in accordance with TriMatrix SOP GR-09-128.

13.2.1 Label all glassware with the sample identification number and other necessary information including whether the extraction is to be a matrix spike (MS), matrix spike duplicate (MSD), blank spike (BS) or extraction blank (BLK).

13.2.2 For the low concentration extraction, perform the following steps rapidly to avoid the loss of compounds from evaporation:

13.2.2.1 For sandy to dry samples, weigh 30 ±0.1 g into a 400 mL beaker and add 20 - 25 g of anhydrous sodium sulfate. Mix well.

13.2.2.2 For wet to gummy samples, weigh 30 ±0.1 g into a 400 mL beaker and add 55 - 60 g of anhydrous sodium sulfate. Mix well.

13.2.2.3 The sample/sodium sulfate mixture should have a free-flowing powder texture after mixing in the sodium sulfate. Add more sodium sulfate as necessary to achieve a free-flowing powder texture.

13.2.2.4 To all extractions, add 1.0 mL of BNA surrogate. However, if concentrating to a final volume of 1.0 mL, only add 100 µL.

13.2.2.5 For matrix and blank spikes, add 1.0 mL of spiking solution. However, if concentrating to a final volume of 1.0 mL, only add 100 µL.

13.2.2.6 Add 100 mL of 1:1 methylene chloride/acetone to each beaker, more may be required if a large amount of sodium sulfate was needed.

13.2.3 Extract by positioning the ¾ inch horn about 1/2 inch below the solvent surface without touching any sample solid or the beaker wall.

13.2.4 Sonicate for three minutes at full power (**OUTPUT CONTROL** knob set at 10 and **PERCENT-DUTY CYCLE** set at 50%). Do not use the microtip horn.

13.2.5 Assemble a K-D flask to receive the extraction solvent as follows:

13.2.5.1 Attach a 10 mL concentrator tube to the K-D flask.

13.2.5.2 Line a 100 mm filter funnel with 24 mm P8 filter paper then add approximately 1 inch of anhydrous sodium sulfate.

13.2.5.3 Place the funnel in the K-D flask.

13.2.5.4 The K-D flask is ready to receive extraction solvent.

13.2.6 From the extraction beaker, decant as much solvent as possible into the K-D flask by pouring through the funnel.

13.2.6.1 If the extraction solvent contains very fine particulate that plugs the P8 filter paper, filter through the vacuum filter instead.

13.2.6.2 Assemble the vacuum filtration apparatus consisting of a 500 mL side-arm flask, 90 mm Buchner funnel and vacuum pump.

13.2.6.3 Filter using the glass fiber filter paper under vacuum then quantitatively transfer from the side-arm flask to the K-D flask for concentration.

13.2.7 Repeat the ultrasonic extraction twice more with additional 100 mL aliquots of 1:1 methylene chloride/acetone, decanting extraction solvent after each sonication.

13.2.8 If after the third sonication, there is no noticeable reduction in solvent color, extract the sample with up to two additional aliquots of methylene chloride/acetone. Record the total number of aliquots used if more than 3.

13.2.9 On the final ultrasonic extraction, decant the solvent then transfer all solid sample from the extraction beaker into the filter funnel and quantitatively rinse with methylene chloride. Let the funnel stop dripping solvent before removing from the K-D flask.

13.2.10 Concentrate extracts as follows:

13.2.10.1 Add one or two clean boiling chips to each concentrator tube and attach a three-ball Snyder column.

13.2.10.2 Pre-wet the Snyder column by adding approximately 1 mL of methylene chloride through the top.

13.2.10.3 Place the assembled concentrator in the water bath (set at 80 - 90° C) so the concentrator tube is partially immersed and the entire lower rounded surface of the K-D flask is bathed with vapor.

13.2.10.4 At the proper rate of distillation, the Snyder column balls will actively chatter but not flood. Until chattering begins, rattle the Snyder column periodically to prevent premature flooding.

13.2.10.5 When the apparent volume of liquid reaches 3 mL (in ten to fifteen minutes), remove the concentrator and let drain for at least 10 minutes.

Note: When the solvent volume is reduced below 1 mL, some analytes will be lost. If extracts are concentrated to dryness or near-dryness, the entire extraction must be repeated.

13.2.11 Quantitatively transfer the concentrate to a 10.0 mL calibrated vial, rinsing the concentrator tube several times with methylene chloride. Transfer all rinsate to the vial. After rinsing, dilute to 10.0 mL with methylene chloride.

13.2.12 Cap each vial with a screw-cap PTFE-lined septum lid and place in the GC/MS refrigerator.

13.2.13 When lower reporting limits are required, concentrate to 1.0 mL final volume using a micro-Snyder column after the initial concentration:

13.2.13.1 Remove the concentrator tube and rinse the ground-glass joint with methylene chloride.

13.2.13.2 Add another clean boiling chip then attach a 2-ball micro-Snyder column.

13.2.13.3 Pre-wet the column with 0.5 mL methylene chloride.

13.2.13.4 Place the concentrator tube in a water bath, partially immersed. Water temperature must be 80 - 90° C. Care must be taken to avoid bumping or column flooding due to immersing the concentrator tube too deep.

13.2.13.5 When the apparent volume reaches 0.5 mL, remove from the bath. Cool for at least ten minutes before removing the Snyder column.

13.2.13.6 Rinse the ground-glass joint with 0.2 mL of methylene chloride after removing the Snyder column.

13.2.13.7 Quantitatively transfer extracts to a 1.0 mL calibrated vial using a clean, disposable, Pasteur pipet, rinsing the concentrator tube with methylene chloride.

13.2.13.8 Adjust the final volume to 1.0 mL then cap tightly and store in the GC/MS refrigerator until analysis.

13.3 Samples expected to contain high concentrations of organics (greater than 20 mg/kg) are extracted based on the following:

13.3.1 Mix samples thoroughly as in Section 13.2.

13.3.2 Gummy, fibrous or oily samples may also need particle size reduction for maximum extraction efficiency. Analyst experience must be used in such cases.

Note: Minimize exposure of the sample to air to prevent analyte loss from evaporation.

13.3.3 Label all glassware with the sample identification number and other necessary information including whether the extraction is to be a matrix spike (MS), matrix spike duplicate (MSD), blank spike (BS) or extraction blank (BLK).

13.3.4 Weigh 2.0 ±0.1 g of sample into a 20 mL vial. Record the weighing.

13.3.5 Add 2 g anhydrous sodium sulfate and mix into a free-flowing powder. If necessary, add more sodium sulfate to achieve the appropriate texture.

13.3.6 Add 1.0 mL of BNA surrogate solution to all extractions.

13.3.7 Add 1.0 mL of BNA spike solution to matrix and blank spikes.

13.3.8 Bring the total solvent volume to 10.0 mL by adding 9.0 mL of methylene chloride to vials when no spike solution is added. Add 8.0 mL of methylene chloride to matrix and blank spikes.

13.3.9 Extract using the 1/8 inch tapered microtip horn for 2 minutes at **OUTPUT CONTROL** set at 5 and **PERCENT-DUTY CYCLE** set at 50%, in pulse mode.

13.3.10 Loosely pack a small glass funnel with fine glass wool. Rinse with methylene chloride. Filter the extract solvent through the glass wool and collect in a 14 mL vial. Do NOT bring to a final volume of 10 mL or rinse the glass funnel with additional solvent as the final volume was previously measured. Cap tightly and store in the GC/MS refrigerator until analysis.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Extraction analysts are responsible for extraction documentation and data integrity. All documentation must be correctly filled in. It is important to document extractions by correctly filling in, turning in and filing accurate paperwork. This is mandatory for quality control and to provide clients with defensible data.

14.2 Report extractions as follows:

14.2.1 Input all extraction data to Element™. Extraction reports must be filled in completely to insure that results are reported correctly and data is associated with the right quality control batch number.

14.2.2 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in completely and correctly.

14.3 Complete all laboratory records as follows:

14.3.1 Logbooks must be filled in completely and correctly. Corrections are to be made with a line-out, not a write-over or scribble-out. Blank lines in the logbook must be Z'd out.

14.3.2 Transfer extraction summary benchsheets (including CoC forms) to the GC/MS analyst with finished extracts.

15.0 QUALITY ASSURANCE

15.1 Extraction blanks must be extracted at the start of each working shift. Blanks are prepared by weighing 30 g of anhydrous sodium sulfate, adding surrogate and extracting by the same procedure used for samples. Extraction blanks extracted at the start of the shift may be used to initiate an extraction batch or up to 20 samples (including the blank spike and matrix spikes) or may be placed within an existing extraction batch having less than 20 samples.

15.2 Surrogate must be added to all extractions, including matrix and blank spikes.

15.3 A blank spike must also be extracted at the start of each working shift. A blank spike is prepared by adding 1.0 mL of spike solution and surrogate to 30 g of anhydrous sodium sulfate then extracting by the same procedure used for samples.

15.4 Extract a matrix spike and matrix spike duplicate, provided sufficient sample is received, at a minimum of every 20 samples or once per week for each matrix. Whichever is more frequent. A Matrix spike is prepared by adding 1.0 mL of spike solution and surrogate to a sample then extracting by the same procedure used for un-spiked samples.

15.5 Perform corrective action for out-of-control quality control extractions in accordance with TriMatrix SOP GR-04-101.

15.6 Refer to TriMatrix SOP GR-04-103 for quality control acceptance limits and additional details.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before extraction of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by completing a successful initial demonstration of capability (IDC) study.

16.2 Prepare four blank spikes by spiking 30 g of anhydrous sodium sulfate with 100 µL of spiking solution to each, with surrogate. Extract as low-concentration samples, going through all extraction steps in the procedure. Concentrate to 1.0 mL.

Note: These IDC blank spikes need to include all BNA analytes.

16.3 After completing the extraction, have the four extracts analyzed in accordance with TriMatrix GR-04-103.

16.4 Input results to the IDC spreadsheet located on the laboratory intranet library to calculate percent recovery and relative standard deviation for each spiked analyte.

16.4.1 For each analyte, percent recovery must be within laboratory established limits and relative standard deviation must be less than or equal to 20%. If all analytes meet acceptance, the

demonstration of capability study is complete and the analyst is authorized to extract samples by this procedure.

16.4.2 If any analyte fails any acceptance limit, locate and correct the problem then repeat the study successfully for the failed analyte.

16.5 Repeated failure indicates a problem with the extraction procedure and/or techniques used. If this occurs, locate the problem, correct the procedure and/or techniques used and repeat the IDC study successfully.

16.6 Samples may not be prepared by the analyst until a successful IDC study has been completed.

16.7 A successful continuing demonstration of capability (CDC) study must be completed annually by all analysts by one of the following approaches:

16.7.1 By repeating the IDC study.

16.7.2 By using four consecutive blank spike results obtained during the course of routine sample extraction and when extracted exclusively by the analyst.

16.7.3 By extracting an acceptable blind performance testing sample.

16.7.4 By exclusively extracting a method detection limit (MDL) study. The last four results of the study may be used.

16.8 A Method Detection Limit (MDL) study must be performed annually for both extraction procedures in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of a laboratory chemical without first referencing appropriate written disposal instructions for that particular material.

17.3 Conserve the use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 2, December, 1996, Method 3550B, "Ultrasonic Extraction"

20.0 ATTACHMENTS

20.1 Extraction Laboratory Soils Logbook Example

20.2 Standards Log Example

Attachment 20.1
Extraction Laboratory Soils Logbook Example

TriMatrix Laboratories, Inc.

Organic Extraction Lab
Soil Extraction Logbook Date: 9-21-07

Client	Project/ Submittal	Sample Number Range	LIMS Batch ID	Test or Parameter	Surrogate Spike Number and Volume		Matrix Spike Number and Volume	
[redacted]	0709361	BLK, BS, MS, MSD 0709360-01, 0709361-01	0710993	3550/8270	7050576	100 mL	7090398	100 mL
"	"	"	5710945	3550/8082	7070018	1000 mL	7070949	500 mL
[redacted]	0709238	0709238-17-18	"	"	"	"	"	"
[redacted]	0709280	0709280-01	"	"	"	"	"	"
[redacted]	0709413	0709413-01	"	"	"	"	"	"
[redacted]	0703646-01	BLK, BS, MSD 0709360-01, 0709361-01	0710946	3550/8081	7070018	1000 mL	709 0892	500 mL

LIMS Sample ID	Bath Temp. °C	Initial Volume (g)	Final Volume (mL)	Analyst Initials
0710993-BLK	70	30.0	1.0	BJH
-BS		30.0		
-MS		30.0		
-MSD		30.0		
0709360-01		30.0		
0709361-01	↓	30.0	↓	↓
0710945-BLK	90	30.0	10.0	BJH
-BS		30.0		
-MS		30.0		
-MSD		30.0		
0709238-17		30.0		
0709280-01		30.0		
0709328-18		30.0		
0709360-01		30.0		
0709361-01		30.0	↓	
0709413-01	↓	15.0	5.0	↓
0710946-BLK	90	30.0	10.0	BJH
-BS		30.0		
-BSD		30.0		
0709360-01		30.0		
0703646-01	↓	30.0	↓	↓

LIMS Sample ID	Bath Temp. °C	Initial Volume (g)	Final Volume (mL)	Analyst Initials

9-21-07

9/21/07

Reagent Information

CH_2Cl_2 Lot #: E134628
1:1 CH_2Cl_2/Acetone Reagent #: PLR 2.32-11
Hexane Lot #: E21E34
Na_2SO_4 Reagent #: PLR 2.3-7
Acetonitrile Lot #: NA
Other #: NA

file: Prep Lab Soil Logbook page: 9 of 20 revision: 0.0

Attachment 20.2
Standards Log Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
7090398

Description:	Short List Spike	Expires:	Oct-31-07
Standard Type:	Analyte Spike	Prepared:	Sep-12-07
Solvent:	MeCl2 Lot#E13468	Prepared By:	Andrea S. Colborn
Final Volume (mls):	50	Department:	Semivolatiles MS
Vials:	1	Last Edit:	Sep-12-07 13:39 by ASC

Analyte	CAS Number	Concentration	Units
1,2,4-Trichlorobenzene	120-82-1	100	ug/mL
1,4-Dichlorobenzene	106-46-7	100	ug/mL
2,4-Dinitrotoluene	121-14-2	100	ug/mL
2-Chlorophenol	95-57-8	150	ug/mL
4-Chloro-3-methylphenol	59-50-7	150	ug/mL
4-Nitrophenol	100-02-7	150	ug/mL
Acenaphthene	83-32-9	100	ug/mL
Naphthalene	91-20-3	100	ug/mL
N-Nitroso-di-n-propylamine	621-64-7	100	ug/mL
Pentachlorophenol	87-86-5	150	ug/mL
Phenol	108-95-2	150	ug/mL
Pyrene	129-00-0	100	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
6110354	(AMP) Acids matrix Spike mix(sh	Nov-09-06	** Vendor **	Feb-28-09	Nov-09-06 15:31 by ASC	1
7090396	Naphthalene Stock	Sep-12-07	Andrea S. Colborn	Sep-12-08	Sep-12-07 13:25 by ASC	0.5
A602407	(AMP)Base/Neutrals Matrix Spike	Feb-13-06	** Vendor **	Jan-31-07	Feb-13-06 11:38 by ASC	1

Reviewed By _____ Date _____

Approved By: _____ 2-12-09 **QA Officer**

Approved By: _____ BSI+ 2/12/09 **Area Supervisor**

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Extraction of Organochlorine Pesticides, PCBs and Chlorinated Hydrocarbons from Water

SW-846 Method 3510C
EPA Method 608
EPA Method 612

APPROVALS:

Area Supervisor: Brian Hall Date: 8/10/09
Brian J. Hall

QA Officer: Tom C. Boocher Date: 8-10-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 8/10/09
Jeff P. Glaser

Procedure Number: GR-09-107
Revision Number: 5.2

Date Initiated: 3/30/94
Effective Date: 8/12/09

Date Revised: 7/27/09
Pages Revised: All

By: Andrea S. Colborn
Total Number of Pages: 22

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

UNCONTROLLED COPY

1.0 SCOPE AND APPLICATION

1.1 This procedure has detailed instructions for extracting organochlorine pesticides and polychlorinated biphenyls and chlorinated hydrocarbons from aqueous samples using methylene chloride as the extraction solvent. The procedure also describes the concentration technique suitable for preparing extracts for analysis.

1.2 This procedure is restricted to use by or under the supervision of a trained analyst. Each analyst must demonstrate the ability to generate acceptable results completion of a demonstration of capability study before actual sample extractions may be performed.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Method 3510C, Revision 3, December 1996, "Separatory Funnel Liquid-Liquid Extraction"

2.2 *40 Code of Federal Regulations*, latest edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, "Organochlorine Pesticides and PCBs"

2.3 *40 Code of Federal Regulations*, latest edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 612, "Chlorinated Hydrocarbons"

3.0 SUMMARY OF PROCEDURE

3.1 A measured volume of sample, usually one liter, is serially extracted at neutral pH with methylene chloride using a 2 L glass separatory funnel.

3.2 Extracts are dried with sodium sulfate and concentrated with Kuderna-Danish concentration glassware.

3.3 Solvent exchange to hexane is performed and the extract is again concentrated to a final volume of 2.0 mL.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to TriMatrix SOP GR-03-120 and SOP GR-03-128 for parameters applicable to this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-03-128, *Gas Chromatography Analysis of Polychlorinated Biphenyls (PCBs)*, latest revision

5.2 TriMatrix SOP GR-03-120, *Gas Chromatography Analysis of Organochlorine Pesticides*, latest revision

5.3 TriMatrix SOP GR-09-109, *Sulfur Cleanup*, latest revision

UNCONTROLLED COPY

5.4 TriMatrix SOP number GR-09-110, *Sulfuric Acid Cleanup*, latest revision

5.5 TriMatrix SOP GR-09-120, *Florisil/Silica Gel Column Cleanup of PCB's, Toxaphene, and Chlordane*, latest revision

5.6 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration,* latest revision

5.7 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.8 TriMatrix SOP GR-16-100, *Glassware Cleaning and Preparation for the Organics Extraction Laboratory*, latest revision

5.9 TriMatrix SOP GR-09-111, *Florisil Column Cleanup,* latest revision

5.10 TriMatrix SOP GR-04-101, *semi-Volatile Organic Laboratory Corrective Actions*, latest revision

5.11 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interferences can be caused by contaminants in solvents, reagents, glassware or sample processing hardware leading to discrete artifacts or elevated baselines. All materials must routinely demonstrate freedom from interferences by analysis of laboratory reagent blanks.

6.1.1 Glassware must be scrupulously cleaned in accordance with TriMatrix SOP GR-16-100.

6.1.2 Use only high purity reagents and pesticides-grade solvents to minimize interferences.

6.2 Matrix interference may be caused by contaminants co-extracted from samples. Matrix interference will vary considerably from matrix to matrix. Such interference can affect analyte recovery and re-extraction or other corrective action may be necessary if surrogates and/or other spiked compounds fail established laboratory recovery limits.

6.3 Phthalate esters cause a direct interference with analyte extractions. Avoid using flexible plastics in contact with solvent to minimize this type of interference.

7.0 SAFETY PRECAUTIONS

7.1 Wear a laboratory coat and approved safety glasses while in the organic extractions laboratory. In addition, disposable gloves must be worn whenever samples or reagents are handled.

7.2 Follow all instructions outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.3 For laboratory waste disposal, refer to TriMatrix SOP GR-15-102.

UNCONTROLLED COPY

7.4 The total toxicity and/or carcinogenicity of reagents used in this procedure have not been precisely defined.

7.4.1 Treat all chemicals as a potential health hazard.

7.4.2 Reduce expose to the lowest possible level by adherence to established safety policies.

7.4.3 Material Safety Data Sheets are maintained on the laboratory intranet of all chemicals used in this procedure. Consult the MSDS for detailed chemical information.

7.5 Samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean contaminated personal protective equipment before using again.

7.6 Bring all safety issues to the attention of the Area Supervisor and/or Health and Safety Officer.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are collected in 1000 mL screw-cap amber glass jars with PTFE-lined lids. No preservative is required.

8.2 All samples must be extracted within seven days of the collection date, and analyzed within 40 days of extraction except for method 608 PCB wastewater samples which have a year to extract.

8.3 When not in use, samples must be stored in the walk-in cooler, at 4 ±2° C.

8.4 Analysts must use care when handling sample containers to avoid loss due to breakage.

8.5 After extraction and/or cleanup, samples must be stored in the GC laboratory refrigerator at 4 ±2° C until analysis.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Erlenmeyer flasks: 300 mL

9.2 Separatory funnels: 2 L glass with PTFE stopcock and PTFE stopper

9.3 Filtering funnels: 100 mm

9.4 Filter paper: Fisher P8, coarse or equivalent

9.5 Kuderna-Danish (K-D) concentrator glassware

9.5.1 Concentrator tubes: 10 mL, graduated (Kontes K-570050-1025 or equivalent)

9.5.2 Concentrator flasks: 500 mL (Kontes K-570001-500 or equivalent)

UNCONTROLLED COPY

9.5.3 Concentrator tube clips

9.5.4 Snyder columns: Three-ball macro (Kontes K-50300-0121 or equivalent)

9.5.5 Snyder columns: Two-ball micro

9.6 Auto-sampler vials: 4.0 mL with PTFE-lined screw cap lids, pre-calibrated to 2.0 mL as outlined in TriMatrix SOP GR-09-106.

9.7 Boiling chips: PTFE, methylene chloride rinsed

9.8 pH paper: wide-range (0-14)

9.9 Phase separation paper

9.10 N-EVAP nitrogen evaporation bath

9.11 Pasteur pipettes: 2 mL disposable, glass

9.12 Water bath: variable temperature, capable of maintaining temperature to ±5° C

9.13 Syringes: 100 µL, 500 µL, 1000 µL

9.14 Graduated cylinders: 1 L

9.15 Analytical Balance, capable of accurately weighing 0.0001 g

9.16 Hot Plate: capable of variable temperature control to ±5° C

9.17 Glass wool

9.18 Volumetric flasks: 10 mL, 50 mL, 100 mL

9.19 Vials: 40 mL amber, with PTFE-lined screw cap lids

9.20 Centrifuge: variable speed

9.21 Centrifuge tubes: 40 mL, PTFE

9.22 Sodium sulfate rinsing equipment:

9.22.1 Buchner funnel: 20 cm

9.22.2 Flask: 4 L Vacuum

9.22.3 Drying pan: 13 x 9 inch, metal

UNCONTROLLED COPY

9.22.4 Vacuum pump

9.22.5 Drying oven

9.22.6 Stopper: PTFE with hole for the Buchner funnel

9.22.7 Clamps: supporting

9.22.8 Filter paper: qualitative fast, 20 cm

9.22.9 Squirt bottle: PTFE

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water

11.1.1 Reagent water is laboratory-purified and methylene chloride extracted (unless otherwise specified) water in which no interferences are present at or above any reporting limit.

11.1.2 All reagent water used to dilute samples and blanks must be pre-extracted with methylene chloride to minimize interference from phthalate ester contamination:

11.1.2.1 Add 1000 mL of laboratory grade water to a 1000 mL separatory funnel

11.1.2.2 Add 60 mL of methylene chloride and extract by vigorously shaking with frequent venting.

11.1.2.3 Let the methylene chloride settle for at least 10 minutes then drain and discard.

11.1.2.4 The remaining mixture is then ready for diluting samples and for use as blanks.

11.2 Methylene chloride: Pesticide grade or equivalent

11.3 Hexane: Pesticide grade or equivalent

11.4 Sodium sulfate: ASC grade, anhydrous granular, rinsed:

11.4.1 This reagent must be rinsed before use. Assemble the sodium sulfate rinsing apparatus and clamp securely.

UNCONTROLLED COPY

11.4.2 Insert filter paper in the Buchner funnel and add the entire contents of a 2.5 kg sodium sulfate container to the funnel.

11.4.3 Add 1 L of methylene chloride to the glass sodium sulfate container, rinsing down the inside with methylene chloride from a PTFE squirt bottle.

11.4.4 Pour this methylene chloride over the sodium sulfate in the Buchner funnel, letting it drain without applying vacuum. Add more methylene chloride if necessary to completely immerse the sodium sulfate.

11.4.5 After most of the solvent has drained, apply vacuum and rinse with an additional 100 mL from the PTFE squirt bottle. Maintain vacuum until solvent stops draining.

11.4.6 Transfer the rinsed sodium sulfate to a drying pan and heat in a drying oven at 120° C for at least one hour.

11.4.7 Remove from the oven with heat-resistant gloves and cool in a hood. Always place a caution sign by the pan while cooling.

11.4.8 After cooling, return to the original container using a glass funnel.

11.4.9 Label the container "Rinsed" with the organic preparation laboratory reagent identification (ID) number then place it in reagent storage.

11.5 Sodium Hydroxide solution (10N): Carefully, dissolve 400 g sodium hydroxide pellets in 1 L of reagent water.

CAUTION: This solution generates significant heat. Add pellets slowly and cool the solution before using.

11.6 Sulfuric acid solution (1:1 v/v): Carefully, add 500 mL concentrated sulfuric acid to 500 mL reagent water in a glass container.

CAUTION: This solution generates significant heat. Add acid slowly to the water and cool before using. NEVER add water to acid.

12.0 STANDARDS PREPARATION

12.1 Laboratory-made surrogates and spike solutions are made by the following rules:

12.1.1 Obtain glassware and materials required for surrogates or spike solutions being made.

12.1.2 Use the appropriate solvent for dilution.

12.1.3 Label all glassware and vials holding surrogate/spiking solutions.

UNCONTROLLED COPY

12.1.4 Use minimal headspace in vials.

12.2 Surrogate and spiking solutions are stored in 40 mL narrow-mouth amber vials, labeled with the following information:

12.2.1 Surrogate or spike name

12.2.2 Laboratory-assigned surrogate or spike ID

12.2.3 Date made

12.2.4 Analyst initials

12.2.5 Solvent

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 Using a microsyringe, inject the stock standard into a volumetric flask approximately half full of the appropriate solvent. For all standards, use only pesticide grade or better solvents. Make sure the syringe tip is below the solvent surface when injecting. Invert enough times to mix thoroughly. All surrogate and spiking standards are stored in 40 mL amber jars with PTFE-lined lids.

12.4 Surrogate/spike standards data is recorded in the extraction standards log. Surrogate/spiking solutions are stored in the organic extractions laboratory refrigerator at 4 ±2°C. A dilution of the spiking solution must be analyzed by the analytical laboratory to verify the concentration prior to use. The concentration recovered must be within 80 – 120%.

12.5 Neat compounds must be ACS grade or better and be weighed with the calibrated analytical balance in the hood. If purity of a neat standard is below 95%, adjust the concentration in all subsequent calculations. Do not use any neat compound past its expiration date. If using the last of a compound, see that it is promptly re-ordered before it runs out.

12.5.1 For neat solids, weigh to the nearest 0.0001 g in a volumetric flask and record the mass. Swirl the flask to mix after adding solvent. If a compound does not dissolve, place the flask in a small sonicator bath to aid in dissolution. Closely monitor the solution and remove as soon as the compound has dissolved. Do not over-sonicate as excessive heat will be generated. Only after complete dissolution, dilute to volume and mix well by inverting at least three times. Discard the volume in the flask neck and transfer the rest to pre-labeled vials with minimal headspace. Store the vials at 4 ±2° C.

12.5.2 For neat liquids, weigh into a tared volumetric flask half-full of the appropriate solvent. Add the neat liquid drop-wise using a Pasteur pipette directly into the solvent, being careful not to touch the inside of the flask neck. Record the mass, swirl to mix then dilute to volume. Mix well and discard the volume in the flask neck. Transfer the rest to pre-labeled vials with minimal headspace and store at 4 ±2° C.

UNCONTROLLED COPY

12.6 The shelf-life of surrogate and spiking solutions is six months for working solutions and one year for stock concentrations. Dispose of any solution sooner if a manufacturer's expiration date occurs within that time for any neat material used. Once a chemical or solution has expired, it must be removed from the laboratory and disposed of. Monitor the expiration date of all chemicals and solutions. To avoid costly rush shipping, it is important to promptly order more chemical once consumed.

12.7 Often when making surrogate and spiking solutions, serial dilutions are required to achieve proper concentration. Serial dilutions are made from a higher concentration. The following example illustrates making a 1.0 mg/L working solution from a 10,000 mg/L stock concentration:

$$10000 \text{ mg/L} \times \frac{1 \text{ mL stock standard}}{100 \text{ mL final volume}} = 100 \text{ mg/L}$$

$$100 \text{ mg/L} \times \frac{1 \text{ mL stock standard}}{100 \text{ mL final volume}} = 1.0 \text{ mg/L}$$

12.8 When making a dilution, all data must be entered into the standards log in Element™ which is the laboratory information management system (LIMS) .

12.8.1 Refer to Attachment 20.2 for an example standards log entry.

12.8.2 Record each serial dilution made from a solution with a full explanation of what was done.

12.8.3 If a dilution of a dilution is made then the resultant solution would be given another Element™ number. This nomenclature is continued for each subsequent dilution.

12.8.4 For solution preparations, use only pesticide-grade or better solvents.

12.8.5 Before using a solution, the calculation performed when calculating concentrations must be verified by a second analyst. Once the calculation has been verified, solution concentration must be verified by actual analysis. If analytical recovery is not within 80 – 120%, the solution may not be used.

12.9 Surrogate solution concentration:

12.9.1 Surrogates are made from purchased solutions containing both surrogates and diluted to 0.20 mg/L. Water samples are spiked with 1.0 mL of the dilution to give extracts the same concentration since final extract volume is 2.0 mL.

Compound(s)	Source	Solvent	Stock Concentration	Stock Dilution	Working Concentration
2,4,5,6-Tetrachloro-m-xylene and Decachlorobiphenyl	Absolute Standards (#20023)	Acetone	200 mg/L	100 µL:100 mL	0.20 mg/L

UNCONTROLLED COPY

12.10 Pesticide analysis spike solution concentration:

12.10.1 Spikes are made from purchased solutions and diluted to 0.20 mg/L. Water samples are spiked with 1.0 mL of the dilution to give extracts the same concentration as surrogates since final extract volume is 2.0 mL.

Compound(s)	Source	Solvent	Stock Conc.	Stock Dilution	Working Conc.
TCL Pesticides	Purchased	Methanol	1000 mg/L	80 µL:100 mL	0.80 mg/L

12.11 PCB analysis spike solution concentration:

12.11.1 Spikes are made from purchased solutions and diluted to 1.0 mg/L. Water samples are spiked with 1.0 mL of the dilution to give extracts a concentration of 0.50 mg/L.

Compound(s)	Source	Solvent	Stock Conc.	Stock Dilution	Working Conc.
PCBs 1016, 1221, 1232, 1242 1248, 1254, 1260	NSI	Acetone	1000 mg/L	1:1000	1.0 mg/L

12.12 Chlorinated Hydrocarbon Spike Solution is made from purchased solution at vary concentrations:

Compound(s)	Source	Stock Conc.	Stock Dilution	Working Conc.
2-Chloronaphthalene	O_2SI	2500	20 µL:25 mL	2 mg/L
1,2,3-Trichlorobenzene	O_2SI	100	21 µL:25 mL	0.08 mg/L
1,2,4,5-Tetrachlorobenzene	O_2SI	100	22 µL:25 mL	0.08 mg/L
1,2,4-Trichlorobenzene	O_2SI	100	23 µL:25 mL	0.08 mg/L
1,2-Dichlorobenzene	O_2SI	2500	24 µL:25 mL	2 mg/L
1,3,5-Trichlorobenzene	O_2SI	100	25 µL:25 mL	0.08 mg/L
1,2,3,4-Tetrachlorobenzene	O_2SI	100	26 µL:25 mL	0.08 mg/L
1,4-Dichlorobenzene	O_2SI	2500	27 µL:25 mL	2 mg/L
Pentachloronitrobenzene	O_2SI	100	28 µL:25 mL	0.08 mg/L
Hexabromobenzene	O_2SI	100	29 µL:25 mL	0.08 mg/L
Hexachlorobenzene	O_2SI	100	30 µL:25 mL	0.08 mg/L
Hexachlorobutadiene	O_2SI	100	31 µL:25 mL	0.08 mg/L
Hexachlorocyclopentadiene	O_2SI	100	32 µL:25 mL	0.08 mg/L
Hexachloroethane	O_2SI	100	33 µL:25 mL	0.08 mg/L
Pentachlorobenzene	O_2SI	100	34 µL:25 mL	0.08 mg/L
1,3-Dichlorobenzene	O_2SI	2500	35 µL:25 mL	2 mg/L

13.0 ANALYTICAL PROCEDURE

UNCONTROLLED COPY

13.1 Assemble the glassware and samples.

13.1.1 Rinse all glassware with methylene chloride prior to use.

13.1.2 Label each separatory funnel, Erlenmeyer flask, K-D concentrator and concentrator tube with the following:

13.1.2.1 The sample ID number

13.1.2.2 The analysis

13.1.2.3 "S" for surrogate addition (Erlenmeyer flasks only)

13.1.2.4 "L" for spike addition, where applicable (Erlenmeyer flasks only)

13.1.2.5 The final extract volume and units

13.2 Determine the sample volume as follows:

13.2.1 Shake all sample jars well. If a shaken sample remains clear, mark the water level on the outside of the jar then transfer its entire contents to a separatory funnel. Do not discard the sample jar. After extraction is complete, fill the jar with tap water to the water level mark (made before the sample was emptied) then measure with a graduated cylinder to attain the sample volume.

13.2.2 If a shaken sample is so cloudy it is difficult to see through the jar, let the sediment settle for approximately five minutes then decant the aqueous portion into a clean graduated cylinder to measure the volume. Shake any sediment remaining in the jar into the trash. DO NOT RINSE A SAMPLE JAR TO REMOVE SEDIMENT OR RESIDUE. After the sample volume has been measured and recorded, transfer the graduated cylinder contents to a separatory funnel.

Note: Any sample sediment suspected of containing PCBs and/or high concentrations of pesticides must be disposed of as hazardous waste in accordance with TriMatrix SOP GR-15-102.

13.2.3 If less than 500 mL of a sample is measured, consult the project chemist before extracting since less than 500 mL will result in elevated reporting limits, which must be pre-approved by the client.

Note: TCLP leachates extracted using 500 mL do not require consultation with the project chemist before extracting.

13.3 Extraction must be done at a pH of 5-9. Check the sample pH using wide-range pH paper by transferring a couple drops of sample onto the pH strip using a Pasteur pipette. Adjust the pH as necessary using sodium hydroxide or sulfuric acid solutions.

13.4 Add 1.0 mL of surrogate solution to each separatory funnel with a 1.0 mL microsyringe.

UNCONTROLLED COPY

13.4.1 Surrogate is added to all extractions.

13.4.2 Do not submerge the microsyringe needle into the liquid. Slowly inject approximately one inch above the liquid's surface.

13.4.3 Do not inject too fast since splashing may occur. If splashing occurs onto the syringe, rinse it internally and externally with methanol. Discard the rinsate.

13.4.4 Cross out the "S" on each corresponding Erlenmeyer as surrogate is added.

13.5 Add 1.0 mL PCB spiking solution to matrix spikes, matrix spike duplicates and blank spikes if the extraction batch is for PCB analysis. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.6 Add 100 µL pesticide spiking solution to matrix spikes, matrix spike duplicates and blank spikes if the extraction batch is for pesticides analysis. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.7 Add 1.0 mL chlorinated hydrocarbons spiking solution to matrix spikes, matrix spike duplicates and blank spikes if the extraction batch is for chlorinated hydrocarbons analysis. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.8 Rinse sample jars and graduated cylinders (when cylinders are used for individual samples) with methylene chloride as follows:

13.8.1 Add 100 mL methylene chloride to each sample jar. Replace the lid (very important).

13.8.2 Shake and vent as necessary.

13.8.3 Pour into the corresponding graduated cylinders and swirl to rinse the sides.

13.8.4 Finally, pour from the graduated cylinder into the separatory funnel containing sample.

13.9 Extract the quality control and samples as follows.

13.9.1 Stopper each separatory funnel and invert. Immediately open stopcock to release pressure.

13.9.2 Close the stopcock, shake and vent again.

13.9.3 Repeat until there is no evidence of pressure.

13.9.4 Shake vigorously for an additional two minutes.

13.9.5 Let the separatory funnel sit for at least ten minutes to allow phase separation.

13.9.6 If an emulsion forms during extraction:

UNCONTROLLED COPY

13.9.6.1 If the emulsion is minor drain the emulsion through phase separation paper into a 300 μL Erlenmeyer flask. When the phase separation paper is dripping slowly, rinse it with 20-30 mL of methylene chloride.

13.9.6.2 If separation does not occur and the emulsion looks like pudding, drain the entire contents of the emulsion into PTFE centrifuge tubes. Centrifuge for four minutes at 4500 rpm. Once centrifuged, transfer the water phase with any intermediary emulsion back into the 2 L funnel. Pour the bottom layer into a 300 mL Erlenmeyer flask.

13.9.7 If emulsion is minimal, drain the methylene chloride (bottom layer) directly into a 300 mL Erlenmeyer flask.

13.10 Repeat the extraction twice more with 60 mL aliquots of methylene chloride. Rinse the sample jars once more with the first of these solvent volumes.

13.11 Transfer the total extract volume to a K-D concentrator as follows:

13.11.1 Quantitatively transfer each extract from the 300 mL flasks through a funnel containing about one inch of sodium sulfate in P8 filter paper into a properly labeled Kuderna-Danish (K-D) concentrators with an attached 10 mL graduated concentrator tube.

13.11.2 Rinse the flasks with several 20-30 mL aliquots of methylene chloride to complete the transfer.

13.12 Concentrate the extracts as follows:

13.12.1 Add one or two clean boiling chips to each concentrator tube and attach a three-ball Snyder column.

13.12.2 Pre-wet the Snyder columns by adding approximately 1 mL of methylene chloride through the top.

13.12.3 Place the concentrators in a water bath (80-90° C) where the concentrator tube is partially immersed and the entire lower rounded surface of the K-D flask is bathed with vapor.

13.12.4 At the proper rate of distillation, balls of each column will actively chatter but not flood. Until chattering begins, rattle Snyder columns periodically.

13.13 Solvent exchange to hexane:

13.13.1 When the apparent volume of liquid reaches 3-5 mL (in ten to fifteen minutes), remove the Snyder column and quickly add 40-60 mL of hexane and two new boiling chips to each concentrator and replace the Snyder column. Add about 10 mL more hexane to the top of the Snyder column. Increase the temperature of the bath to 90-95° C. Concentrate again to an apparent volume of 5 mL. Remove and cool for at least 10 minutes. The volume in the

UNCONTROLLED COPY

concentrator tube after cooling should be approximately 8-10 mL and appropriate for micro-concentration.

13.14 Micro-Snyder column technique is used to adjust the final extract volume to 2.0 mL. However, if using nitrogen blowdown instead of the micro-Snyder column, proceed to Section 13.4.2.

13.14.1 Micro-Snyder Technique:

13.14.1.1 Remove each concentrator tube and rinse the ground-glass joint with hexane.

13.14.1.2 Add another clean boiling chip then attach a 2-ball micro-Snyder column.

13.14.1.3 Pre-wet the column with 0.5 mL hexane.

13.14.1.4 Place concentrator tubes in a water bath, partially immersed. The water temperature should be 90-95° F. Care should be taken to avoid bumping or column flooding due to immersing too deep.

13.14.1.5 When the apparent volume reaches 1 mL, remove from the bath. Cool for at least ten minutes.

13.14.1.6 Rinse the ground-glass joints with 0.2 mL hexane after removing the Snyder column.

13.14.1.7 Transfer each extract to a 2.0 mL calibrated vial using a clean, disposable, Pasteur pipette.

13.14.1.8 Using approximately 0.8 mL of hexane, rinse each concentrator tube and transfer to the vial.

13.14.1.9 Adjust the final volume to 2.0 mL then cap tightly.

13.14.1.10 Extracts are ready for cleanup or analysis. Store extracts to be cleaned up in the refrigerator.

13.14.2 If using nitrogen blowdown instead of the micro-Snyder column, proceeed as follows:

13.14.2.1 Position sample bottles on the N-EVAP.

13.14.2.2 With a gentle stream of nitrogen, concentrate extracts to <u>not less than</u> 2.0 mL.

13.14.2.3 Extracts are ready for cleanup or analysis. Store extracts to be cleaned up in the refrigerator.

13.15 Extract cleanup

UNCONTROLLED COPY

13.15.1 If necessary, pesticide extracts may be cleaned up using Florisil columns (1000 mg or 20 g) in compliance with TriMatrix SOP GR-09-111.

13.15.2 All PCB extracts must be cleaned up as follows:

13.15.2.1 Sulfuric acid cleanup in compliance with TriMatrix SOP GR-09-110.

13.15.2.2 Copper powder to remove elemental sulfur, sulfur cleanup in compliance with TriMatrix SOP GR-09-109.

13.15.3 If PCB extracts have color and/or do not appear to be free of contaminants after sulfuric acid and copper, then the extracts need cleaned up as follows and in the order listed:

13.15.3.1 Florisil/Silica gel column cleanup in compliance with TriMatrix SOP GR-09-120.

13.15.3.2 Sulfuric acid cleanup in compliance with TriMatrix SOP GR-09-110.

13.15.3.3 Sulfur cleanup in compliance with TriMatrix SOP GR-09-109.

13.16 Store cleaned up extracts at 4 ±2° C in the GC refrigerator.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Extraction analysts are responsible for extraction documentation and data integrity. All documentation must be correctly filled in. It is important to document extractions by correctly filling in, turning in and filing accurate paperwork. This is mandatory for quality control and to provide clients with defensible data.

14.2 Report extractions as follows:

14.2.1 Input all extraction data to Element™. Extraction reports must be filled in completely to insure that results are reported correctly and data is associated with the right quality control batch number.

14.2.2 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in completely and correctly.

14.3 Complete all laboratory records as follows:

14.3.1 Logbooks must be filled in completely and correctly. Corrections are to be made with a line-out, not a write-over or scribble-out. Blank lines in the logbook must be Z'd out.

14.3.2 Transfer extraction summary benchsheets (including CoC forms) to the GC/MS analyst with finished extracts.

15.0 QUALITY ASSURANCE

UNCONTROLLED COPY

15.1 An extraction blank (BLK) and blank spike (BS) must be done daily or for each extraction batch of up to 20 samples, whichever comes first, to demonstrate that extraction interferences are under control.

15.2 Matrix spikes (MS) and matrix spike duplicates (MSD) are extracted at a minimum of each extraction batch of up to 20 samples for analysis group or once a week, whichever is more frequent and provided enough sample is received. However, matrix spikes (MS) and matrix spike duplicates (MSD) are extracted at a minimum of each extraction batch of up to 10 samples for method 608 (primarily wastewater effluent samples) or once a week, whichever is more frequent and provided enough sample is received.

15.2.1 Performance records must be maintained to document the data quality generated.

15.2.2 Matrix spikes are extracted by measuring 1.0 mL of PCB spike solution (or 100 µL of pesticides spike solution) in addition to 1.0 mL of surrogate, to 1 L of sample (or 1.0 mL 8121 spiking solution).

15.3 Blank spikes (BS) are prepared by measuring 1.0 mL of PCB spike solution (or 100 µL of pesticides spike solution) in addition to 1.0 mL of surrogate, into 1 L of organic-free laboratory reagent water (or 1.0 mL 8121 spiking solution).

15.4 Matrix interference from samples or from laboratory-induced contamination may affect analyte recovery. Investigation in accordance with TriMatrix SOP GR-04-101 may be necessary if surrogate and/or spiked compounds fail laboratory-established control limits.

15.5 Surrogates must be added to all extractions.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by successfully completing an Initial Demonstration of Capability (IDC) study. A Continuing Demonstration of Capability (CDC) is required annually.

16.1.1 Initial Demonstration of Capability

16.1.1.1 Prepare a spiking solution at analyte concentrations listed in Section 12.0. Prepare the spiking solution separately from the instrumental calibration standards. Spike 1.0 mL (100 µL for pesticides) of the solution into four, 1 L aliquots of water and extract following all steps in this procedure. After extracting, have the extracts analyzed then input the results to the IDC spreadsheet located on the laboratory intranet library. Average percent recovery as calculated from the spreadsheet must fall within the blank spike acceptability window listed in Element™. Standard deviation of the average recovery must be ≤20%.

16.1.1.2 If the study fails for either recovery or percent difference, locate and correct the source of the problem and repeat the study. If the second study passes, the analyst has demonstrated the procedure successfully. Repeated failure however, will

UNCONTROLLED COPY

indicate a procedure or technique problem. If this occurs, correct the technique or procedure and repeat the study successfully.

16.1.1.3 Samples may not be processed by any analyst or on any instrument until an IDC study has been successfully completed. Copies of successful demonstrations of capability spreadsheets and raw data must be provided to Quality Assurance (QA) for documentation of training.

16.1.2 Continuing Demonstration of Capability (CDC)

16.1.2.1 Annually, a CDC is required by each analyst. The CDC may be accomplished by repeating the IDC study, using the last four results from an exclusively run MDL study or by extracting a successful blind PT study sample. Input results to the same spreadsheet used for IDC studies.

16.2 A Method Detection Limit (MDL) study is required annually in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Method 3510C, Revision 3, December 1996, "Separatory Funnel Liquid-Liquid Extraction"

UNCONTROLLED COPY

19.2 *40 Code of Federal Regulations*, most current edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, "Organochlorine Pesticides and PCBs"

19.3 *40 Code of Federal Regulations*, most current edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 612, "Chlorinated Hydrocarbons"

20.0 ATTACHMENTS

20.1 Preparation Batch Report Example

20.2 Extraction Standards Log Entry Example

20.3 Organic Preparation Laboratory Waters Logbook Example

20.4 Addendum to the Procedure in Reference to Sample Batching

UNCONTROLLED COPY

Attachment 20.1
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0703579 Page 1 of 1

Printed: 4/9/2007 4:54:47PM

Semivolatiles GC, Waste Water, 608 Liquid/Liquid Extraction

Surrogate #1 = 7010436 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0704032	608 PCBs (std 7 aroclors)	0704054	608 PCBs (master list)	0704064	608 PCBs ([redacted])
0704064	608 PCBs (std 7 aroclors)	0704064	608 PCBs (master list)	0704071	608 PCBs ([redacted])
0704081	608 PCBs (std 7 aroclors)				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (mL)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0703579-BLK1		Apr-06-07 13:00	BJH	1000	2	1000				BLANK	
0703579-BS1		Apr-06-07 13:00	BJH	1000	2	1000		7010798	1000	LCS	
0703579-MS1		Apr-06-07 13:00	BJH	1020	2	1000	0704064-02	7010798	1000	MATRIX SPIKE	
0703579-MSD1		Apr-06-07 13:00	BJH	1020	2	1000	0704064-02	7010798	1000	MATRIX SPIKE DUP	
0704032-01	A	Apr-06-07 13:00	BJH	1060	2	1000				[redacted]	report MPB at 0.1ug/L, plz add for TOT
0704054-02	B	Apr-06-07 13:00	BJH	1000	2	1000				[redacted]	[redacted]
0704064-01	C	Apr-06-07 13:00	BJH	1010	2	1000				[redacted]	
0704064-02	F	Apr-06-07 13:00	BJH	1020	2	1000				[redacted]	
0704064-02	F	Apr-06-07 13:00		1020	2	1000				[redacted]	Added for BatchQC in: 0703579
0704064-02	F	Apr-06-07 13:00		1020	2	1000				[redacted]	Added for BatchQC in: 0703579
0704064-04	E	Apr-06-07 13:00	BJH	1060	2	1000				[redacted]	
0704071-01	B	Apr-06-07 13:00	BJH	1020	2	1000				[redacted]	1254 only
0704081-01	C	Apr-06-07 13:00	BJH	1030	2	1000				[redacted]	
0704081-02	D	Apr-06-07 13:00	BJH	1030	2	1000				[redacted]	

Comments:	Analyst Initials:

bch_TriMatrix.rpt

Approved By: [signature] 8-10-09 **QA Officer**

Approved By: BJH 8/10/09 **Area Supervisor**

UNCONTROLLED COPY

Attachment 20.2
Extraction Standards Log Entry Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
7040565

Description:	1016/1260 Soil Spike	Expires:	Oct-16-07
Standard Type:	Analyte Spike	Prepared:	Apr-16-07
Solvent:	Hexane Lot #E08E21	Prepared By:	Brian J. Hall
Final Volume (mls)	100	Department:	Semivolatiles GC
Vials:	1	Last Edit:	Apr-16-07 15:21 by BJH

Analyte	CAS Number	Concentration	Units
PCB-1016	12674-11-2	10	ug/mL
PCB-1016 [2C]	12674-11-2	10	ug/mL
PCB-1260	11096-82-5	10	ug/mL
PCB-1260 [2C]	11096-82-5	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
A604465	(AMP) Aroclor 1016	May-12-06	** Vendor **	Sep-30-08	Apr-12-07 13:29 by JLW	1
A604466	(AMP) Aroclor 1260 stock	Oct-31-05	** Vendor **	Oct-31-08	Apr-12-07 13:35 by JLW	1

Reviewed By _____ Date _____

Page 1 of 1

UNCONTROLLED COPY

Attachment 20.3
Organic Preparation Laboratory Waters Logbook Example

TriMatrix Laboratories, Inc.

Organic Extraction Lab
Water Extraction Logbook

Date: 4/9/07

Client	Project/ Submittal	Sample Number Range	LIMS Batch ID	Test or Parameter	Surrogate Spike Number and Volume		Matrix Spike Number and Volume	
[redacted]	0704064	BLK, BS, MS, MSD 0704064-01-04	0703613	3510/8270	6120791	100 ul	7030610	100 ul
[redacted]	0704095	0704095-05-07, 09	"	"	"	"		
[redacted]	0704100	0704100-01	"	"	"	"		
[redacted]	0703391	BLK, BS, 0703391-06RE1	0703180	8151	A61008	100 ul	7030846	500 ul
ASC 4/9/07								

LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials
0703613BLK	70	1000	1.0	BJH
0704064-01		1010		
0704064-02		1050		
0704064-03		1060		
0704064-04		1060		
0703613MS		1050		
0703613MSD		1050		
0703613BS	↓	1000	↓	↓
0704095-05	70	1060	1.0	BJH
0704095-06	↓	1010	↓	↓
0704095-07		920		
0704095-09	↓	970	↓	↓
0704100-01	70	1060	1.0	BJH
0703182BLK2	68	1000	5.0	ASC
0703174RE1	↓	1030	↓	↓
0703182BS2	↓	1000	↓	↓
		ASC 4/9/07		

LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials
		ASC 4/9/07		

Reagent Information

CH_2Cl_2 Lot #: C40467
Hexane Lot #: C50E54
Na_2SO_4 Reagent #: PLR2.3-2
1:1 H_2SO_4 Reagent #: PLR261-15
1:1 HCl Reagent #: NA
10N NaOH Reagent #: 7040183
NaCl Reagent #: NA

file: Prep Lab Water Logbook page: 1 of 30 revision: 0.0

UNCONTROLLED COPY

Approved By: [signature] 8-10-09 QA Officer

Approved By: BJH 8/10/09 Area Supervisor

Attachment 20.4
Addendum to the Procedure in Reference to Sample Batching

Some clarification of method QC batching requirements and refinement of TriMatrix batching policies is necessary.

It is not permissible to extract more than 20 samples of the same method and matrix without the extraction of additional matrix QC samples.

To prevent this from happening, only one QC batch of a given method and matrix may be open at any one time. When reaching 20 samples or when a Level 3 project arrives, close the currently active batch and begin a new one. It does not matter if the current batch contains less than 20 samples. This policy will be applicable to all extracted samples with 2 exceptions:

#1) If multiple Level 3 and above clients come in on the same day, all of which have samples of the same matrix/method combination, it will be necessary to have more than one QC batch open. At the end of the day, close all the open batches except the one with the fewest samples. That one will remain open as the active batch.

#2) Certain clients send in samples over an extended time period. They also specify which of their samples are to be spiked. The selected sample does not always come in with the first round of samples received. For these clients and under these circumstances it will also be acceptable for multiple QC batches for the same method and matrix are open concurrently. Treat these batches as if they did not exist with regards to batching other samples.

Additionally, except for #2 above, batches may not be held open for longer than 7 days. After seven days close the active batch and begin a new one.

UNCONTROLLED COPY

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Extraction of Organochlorine Pesticides and Polychlorinated Biphenyls from Soil, Sludge and Wipe Samples

SW-846 Method 3550C

APPROVALS:

Area Supervisor: Brian Hall Date: 3/2/09
Brian J. Hall

QA Officer: Tom C. Boocher Date: 2-27-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 3/3/09
Jeff P. Glaser

Procedure Number: GR-09-108
Revision Number: 4.3

Date Initiated: 3/30/94
Effective Date: 3/27/09

Date Revised: 2/26/09
Pages Revised: All

By: Tom C. Boocher
Total Number of Pages: 21

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the extraction of chlorinated pesticides and polychlorinated biphenyls (PCB) from soil, sludge, and wipes.

1.2 Sonication is used to ensure sufficient contact with extraction solvent and facilitate acceptable analyte recovery from most sample matrices. Sonication is not appropriate where extraction efficiencies for low analyte concentrations are required.

1.3 If necessary and when appropriate, extracts will be cleaned up after extraction and concentration.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, February, 2007, Method 3550C, "Ultrasonic Extraction"

3.0 SUMMARY OF PROCEDURE

3.1 Extraction for analyte concentration at or below 20 mg/kg.

3.1.1 Anhydrous sodium sulfate is mixed with 30 g of sample to form a free-flowing solid.

3.1.2 Samples are serially extracted three times by sonication with 1:1 methylene chloride/acetone. Extracts are concentrated then solvent exchanged to hexane. Extracts are concentrated again to 10.0 mL for cleanup and/or GC analysis.

3.1.3 This technique is for low to medium analyte concentrations and negligible matrix interference. If necessary, final extract volumes may be greater than 10 mL or the following technique used.

3.2 Extraction procedure for analyte concentration above 20 mg/kg or difficult sample matrices

3.2.1 Samples with a history of high analyte concentration or matrix interference are extracted using 2.0 g of sample. Extraction analyst discretion based on experience is used to determine the need for this technique.

3.2.2 Anhydrous sodium sulfate is added to form a free-flowing solid.

3.2.3 Samples are extracted once by sonication with hexane. Extracts are filtered through glass wool and collected in a 15 mL vial for cleanup and/or gas chromatographic (GC) analysis.

3.3 Extraction procedure for wipes

3.3.1 Wipes are serially extracted three times by sonication with hexane in the sample container.

3.3.2 The hexane extract is concentrated to 10.0 mL for cleanup and/or GC analysis.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to TriMatrix SOP GR-03-120 for an organochlorine pesticide list associated with this procedure.

4.2 Refer to TriMatrix SOP GR-03-128 for a polychlorinated biphenyl (PCB) list associated with this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-03-128, *Semi-Volatile Laboratory Gas Chromatography Analysis of Polychlorinated Biphenyls (PCBs)*, latest revision

5.2 TriMatrix SOP GR-03-120, *Semi-Volatile Laboratory Gas Chromatography Analysis of Organochlorine Pesticides*, latest revision

5.3 TriMatrix SOP GR-09-109, *Sulfur Cleanup*, latest revision

5.4 TriMatrix SOP GR-09-110, *Sulfuric Acid Cleanup*, latest revision

5.5 TriMatrix SOP GR-09-111, *Florisil® Column Cleanup*, latest revision

5.6 TriMatrix SOP GR-09-120, *Florisil®/Silica Gel Column Cleanup of PCBs, Toxaphene and Chlordane*, latest revision

5.7 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.8 TriMatrix SOP GR-09-128, *Soil Mixing and Grinding,* latest revision

5.9 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration,* latest revision

5.10 TriMatrix SOP GR-16-100, *Equipment Cleaning and Preparation for the Organic Extraction Laboratory,* latest revision

5.11 TriMatrix SOP GR-04-101, *Semi-Volatiles Laboratory Quality Control Corrective Actions*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interference can be caused by contaminants in solvents, reagents, glassware, or sample processing equipment, leading to discrete artifacts or elevated baselines. All materials used in this procedure must routinely demonstrate to be free from interference by analysis of blank spikes (BS).

6.2 Use only reagent-grade or better reagents, and pesticide-grade or better solvents. Clean all glassware in accordance with TriMatrix SOP GR-16-100.

6.3 Phthalate esters cause interference with pesticide and PCB analysis. Avoid using flexible plastics in contact with solvent to minimize this type of contamination.

6.4 Matrix interferences can affect analyte recovery. Repeating an extraction and/or cleanup may be necessary if surrogates or other spiked compounds fail laboratory established control limits.

7.0 SAFETY PRECAUTIONS

7.1 Extraction personnel must wear laboratory coats and approved safety glasses while in the organic extractions laboratory area. Also, disposable gloves must be worn whenever samples, reagents and/or solvents are handled.

7.2 Follow all instructions outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.3 For waste disposal refer to TriMatrix SOP GR-15-102.

7.4 The toxicity and/or carcinogenicity of chemicals used in this procedure has not been fully defined.

7.4.1 Treat all chemicals as a potential health hazard.

7.4.2 Reduce exposure to the lowest possible level by adherence to established safety practices.

7.4.3 A Material Safety Data Sheet (MSDS) is located on the laboratory intranet for all chemicals used in this procedure.

7.5 Bring safety issues to the attention of the Health and Safety Officer and Area Supervisor.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are collected in 60 mL, 125 mL or 250 mL wide-mouth glass jars with PTFE-lined screw-cap lids.

8.2 All samples must be extracted within fourteen days of the collection date and analyzed within 40 days of extraction.

8.3 When not in use, samples must be stored in the walk-in cooler at 4 ±2° C.

8.4 Use care when handling sample containers to avoid sample loss due to breakage.

8.5 After extraction and/or cleanup, store extracts in the GC refrigerator at 4 ±2° C until analysis.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Beakers, heavy-duty Pyrex: 400 mL and 600 mL

9.2 Ultrasonic disrupter:

9.2.1 Fisher model 550 with ¾ inch horn

9.2.2 For the high concentration extraction, use the 1/8 inch tapered microtip attached to a ½ inch horn

9.2.3 Sonabox, for hearing protection

9.3 Kuderna-Danish (K-D) concentrator glassware:

9.3.1 Concentrator tubes: 10 mL, graduated (Kontes K-570050-1025 or equivalent)

9.3.2 Concentrator flasks: 500 mL (Kontes K-570001-500 or equivalent)

9.3.3 Concentrator tube clips

9.3.4 Snyder columns: Three-ball macro (Kontes K-50300-0121 or equivalent)

9.3.5 Snyder columns: Two-ball micro

9.4 Water bath: Variable temperature

9.5 Balance, top-loading, capable of accurately weighing to the nearest 0.01 g

9.6 Spatula, wooden tongue depressors

9.7 Pasteur pipets, disposable, 2 mL

9.8 Boiling chips, PTFE, methylene chloride rinsed

9.9 Vials (14 mL), with PTFE-lined screw-cap lids, calibrated to 10 mL

9.10 Filter paper, qualitative: Fisher P8

9.11 N-EVAP concentrator

9.12 Syringes, micro: 100 uL, 500 µL, and 1000 µL

9.13 Aluminum foil

9.14 Filtration apparatus

9.14.1 Filter flask: 500 mL side-arm

9.14.2 Buchner funnel, 90 mm

9.14.3 Stopper: PTFE, holed

9.14.4 Glass-fiber filter paper for clay samples or samples with fine particulate

9.15 Filter funnel: 100 mm, ribbed

9.16 Sodium sulfate rinsing equipment:

9.16.1 Buchner funnel: 20 cm

9.16.2 Flask: 4 L vacuum

9.16.3 Drying pan: 13 x 9 inch, metal

9.16.4 Vacuum pump

9.16.5 Drying oven

9.16.6 Stopper, PTFE with hole for the Buchner funnel

9.16.7 Clamps, supporting

9.16.8 Filter paper, qualitative fast, 20 cm

9.16.9 Squirt bottle, PTFE, labeled with contents

9.17 Hot Plate: capable of variable temperature control to within ±5° C

9.18 Volumetric Flasks: 10 mL, 50 mL, 100 mL, 1 L

9.19 Amber vials, with PTFE-lined screw-cap lids, 40 mL

9.20 Analytical balances, capable of accurate measurement to the nearest 0.0001g

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Disrupters must be tuned at the beginning of each shift.

10.2 Ultrasonic probes must be cleaned before extracting each new sample and thoroughly cleaned at the end of each shift.

11.0 CHEMICALS AND REAGENTS

11.1 Methylene chloride/acetone, 1:1 (v/v): pesticides grade or better

11.2 Methylene chloride: pesticides grade or better

11.3 Sodium sulfate: ASC grade, anhydrous granular, rinsed:

11.3.1 This reagent must be rinsed before use. Assemble the sodium sulfate rinsing apparatus and clamp securely.

11.3.2 Insert filter paper in the Buchner funnel and add the entire contents of a 2.5 kg sodium sulfate container to the funnel.

11.3.3 Add 1 L of methylene chloride to the container, rinsing down the inside with methylene chloride from a PTFE squirt bottle.

11.3.4 Pour this methylene chloride over the sodium sulfate in the Buchner funnel, letting drain without applying vacuum. Add more methylene chloride if necessary to completely immerse the sodium sulfate.

11.3.5 After most of the solvent has drained, apply vacuum and rinse with an additional 100 mL from the PTFE squirt bottle. Maintain vacuum until solvent stops draining.

11.3.6 Transfer the rinsed sodium sulfate to a drying pan and heat in a drying oven at 120° C for at least one hour.

11.3.7 Remove from the oven with heat-resistant gloves and cool in a hood. Always place a caution sign by the pan while cooling.

11.3.7 After cooling, return to the original container using a glass funnel.

11.3.8 Label the container "Rinsed" with date and analyst's initials then place in reagent storage.

11.4 Hexane: Pesticides grade or better

11.5 Acetone: Pesticides grade or better

11.6 Each lot of methylene chloride, hexane and acetone must be tested before use to show freedom from interference. Submit test results to the quality assurance department for scanning into the certification of analysis file.

12.0 STANDARDS PREPARATION

12.1 Surrogates and spike solutions prepared from neat materials are made by the following rules:

12.1.1 Obtain an analytical balance that weighs to 0.0001 g. Be sure to record mass to the nearest 0.0001 g.

12.1.2 Place the balance in a fume hood and re-calibrate it.

12.1.3 Obtain glassware and materials required for surrogates or spike solutions being made.

12.1.4 Use the required solvent for dilutions.

12.1.5 Minimize headspace in storage vials.

12.2 Store surrogate and spiking solutions in 40 mL narrow-mouth amber vials, labeled with the following information:

12.2.1 Surrogate or spike name

12.2.2 Laboratory-assigned surrogate or spike ID

12.2.3 Date made

12.2.4 Analyst initials

12.2.5 Solvent

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 If solid surrogate or spike compounds are weighed into a volumetric flask and the mass recorded, add enough solvent to dissolve then dilute to volume and invert several times to mix thoroughly.

Note: If solids do not dissolve, place in a small ultrasonic bath to aid in mixing. Do not leave for an extended time because the bath generates heat. After sonication, dilute to volume then transfer to a tightly capped pre-labeled vial and store at 4 ±2° C.

12.4 Weigh liquid surrogate or spike compounds into volumetric flasks containing approximately half-volume of solvent. Use a Pasteur pipet to add compound drop-wise, directly into the solvent. Be careful not to touch the inside of the flask or the solvent surface with the pipet tip. Record the mass and dilute to volume. Invert enough times to mix thoroughly then transfer to pre-labeled, tightly capped vials and store at 4 ±2° C.

12.5 Record surrogate/spike data in an extraction standards log (Attachment 20.1). Store surrogate/spiking solutions in the refrigerator at 4 ±2° C. A dilution of spiking solution must be analyzed by GC/MS and/or GC to check concentration prior to use by the extractions laboratory. Concentration must be within 80 – 120% and hardcopy submitted to the quality assurance department.

12.6 Neat compounds must be ACS grade or better. If the purity of a neat standard is below 95%, the concentration must be accounted for in all subsequent analytical calculations. Do not use any neat compound past its expiration date. If using the last of a compound, promptly re-order.

12.7 Shelf-life of surrogate and spiking solutions is six months for working solutions and one year for stock concentrations. Solutions will be disposed of sooner however if a manufacturer's expiration date occurs within that time for any neat material used. Once a chemical or solution has expired, remove from the laboratory for disposal. Monitor expiration dates of all chemicals and solutions. To avoid costly shipping, it is important to promptly order more chemical once consumed.

12.8 When making surrogate and spiking solutions, serial dilutions are often required to achieve the proper concentration. Serial dilutions are made from a higher concentration. The following example illustrates making a 1.0 mg/L working solution from a 10,000 mg/L stock concentration:

12.8.1 Using a micro-syringe, inject 1.0 mL of 10,000 mg/L solution into a 100 mL volumetric flask approximately half full of the appropriate solvent. Make sure the syringe needle tip is below the solvent surface. Fill to the mark with solvent then cap the flask and invert enough times to mix thoroughly. Concentration in the flask is 100 mg/L.

12.8.2 Inject 1.0 mL of the 100 mg/L solution into another volumetric flask half filled with solvent after rinsing the syringe thoroughly with clean solvent. Fill to the mark with solvent then cap the flask and invert enough times to mix thoroughly. Concentration in this second flask is 1.0 mg/L.

12.8.3 Concentration in the second flask is determined by the following calculation:

$$10000\ \text{mg/L} \times \frac{1\ \text{mL stock standard}}{100\ \text{mL final volume}} = 100\ \text{mg/L}$$

$$100\ \text{mg/L} \times \frac{1\ \text{mL stock standard}}{100\ \text{mL final volume}} = 1.0\ \text{mg/L}$$

12.9 When making a dilution, all data must be entered into the laboratory information management system (Element™). Enter each subsequent dilution in a dilution series separately. Use only pesticide grade (or better) solvents to dilute with.

12.10 Before using a laboratory-prepared solution, verify the calculations used by a second analyst. Once calculations have been verified, verify the solution concentration by actual analysis. If analytical recovery is not within laboratory established acceptance limits, a prepared solution cannot be used. Acceptance limits are 80 – 120% of expected value.

12.11 Dispose of vials containing 5 mL or less of a prepared solution at the end of the shift to minimize contamination and analyte concentration. When not in use, keep spike solution vials in the organic extractions refrigerator.

12.12 Surrogates are normally made from purchased solutions and diluted in acetone to make one 200 mL volume. Check by GC before using. If all surrogates in a spiking solution are within 80-120% of the expected value, the solution is approved for use. Remaining individual surrogate stock solutions are used to calibrate the GC.

Surrogate	Purchased Concentration (mg/L)	Stock Dilution (mL:mL)	Final Working Concentration (mg/L)
2,4,5,6-Tetrachloro-m-xylene	200	1:200	1
Decachlorobiphenyl	200	1:200	1

12.13 Pesticide analysis spike solution concentration:

12.13.1 Spikes are made from purchased solutions and diluted to 1.0 mg/L in hexane. Samples are spiked with 500 µL of the dilution to give extracts the same concentration as surrogates since final extract volume is 10.0 mL.

Pesticide Stock	Purchased Concentration	Dilution	Final Concentration
Purchased Mixture	1000 mg/L	80 µL/100 mL	0.8 mg/L

12.14 PCB spiking solutions are made from individually purchased Aroclors and diluted in hexane to make 100 mL working solutions, which are checked by GC before using. If the Aroclor in a working solution is within 80 - 120% of expected value when tested, the solution is approved for use. Stock solutions are also used to calibrate the GC. PCB spiking solutions are prepared by the GC analyst.

Aroclor	Purchased Concentration (mg/L)	Stock Dilution (mL:mL)	Final Concentration (mg/L)
1221	1000	1:100	10
1242	1000	1:100	10
1248	1000	1:100	10
1254	1000	1:100	10
1260	1000	1:100	10

13.0 ANALYTICAL PROCEDURE

13.1 The Fisher Model 550 ultrasonic disrupter must be tuned at the beginning of each shift, before samples are processed:

13.1.1 Turn the **OUTPUT CONTROL** knob counterclockwise to zero.

13.1.2 Press the **POWER SWITCH** to **ON** (up position). The switch will light up.

13.1.3 When the prompt appears, press **TUNE**. The display will read: [**TUNING - - - PROBE ACTIVE**].

13.1.4 Turn the **OUTPUT CONTROL** knob towards setting 10 (5 if using a microtip).

13.1.4.1 Note the position of the Bar Graph on the display screen. Do NOT exceed 70%.

13.1.4.2 Rotate the **OUTPUT CONTROL** knob clockwise or counterclockwise until a minimum (not maximum) reading is obtained. If a reading of less than 20% cannot be obtained, there is a problem with the disrupter and it must be repaired before using.

13.1.5 Press the **TUNE** key to display prompts for pulse or continuous operation and set the disrupter for pulse operation.

13.1.6 The disrupter is now tuned. Record all tuning information in the disrupter tuning logbook (Attachment 20.2).

13.2 Prior to sonication, homogenize all samples in accordance with the instructions outlined in TriMatrix SOP GR-09-128.

13.3 Label all glassware with sample number and other necessary information including MS, MSD, BS or BLK designations.

13.4 The low concentration extraction is as follows:

13.4.1 Perform the following steps rapidly to avoid loss of more volatile compounds

13.4.2 For sandy to dry samples, weigh 30 ±0.1 g of sample into 400 mL beakers and add 20 g to 25 g of anhydrous sodium sulfate. Mix well.

13.4.3 For wet to gummy samples, weigh 30 ±0.1 g of sample into 400 mL beakers and add 55 g to 60 g of anhydrous sodium sulfate. Mix well.

13.4.4 Samples should have a sandy texture after adding sodium sulfate. Add more sodium sulfate as necessary to achieve this sandy texture.

13.4.5 Measure 1.0 mL of surrogate into all samples and quality control extractions.

13.4.6 For matrix and blank spikes, add 0.5 mL of pesticides or 0.5 mL of PCB spiking solution.

13.4.7 Measure 100 mL of 1:1 methylene chloride/acetone into the beaker.

13.4.8 Sonicate by positioning the 3/4 inch sonication horn about 1/2 inch below the solvent surface without touching any sample solid or the beaker wall.

13.4.9 Sonicate for three minutes at full power (**OUTPUT CONTROL** knob set at 10 and **PERCENT-DUTY CYCLE** set at 50%). Do not use the microtip horn.

13.4.10 Next, assemble a K-D flask for each sample extracted to receive the extraction solvent.

13.4.11 Attach a 10 mL concentrator tube to the flask bottom.

13.4.12 Line 100 mm filter funnels with P8 filter paper then add approximately 1 inch of anhydrous sodium sulfate.

13.4.13 Decant the extraction solvent into the K-D flask through the funnel. Repeat with a different K-D setup for each sample extracted.

13.4.14 If a sample contains very fine particulate that plugs the filter paper, reweigh the sample and filter with a vacuum filter instead.

13.4.14.1 Assemble the vacuum filtration apparatus consisting of a 500 mL side-arm flask, 90 mm Buchner funnel and vacuum pump.

13.4.14.2 Filter using glass fiber filter paper under vacuum and transfer to a K-D flask for concentration.

13.4.15 Repeat the extraction twice more with additional 100 mL aliquots of 1:1 methylene chloride/acetone, decanting off the extraction solvent after each sonication. Repeat for each sample extracted until each sample has been extracted three times and the extract filtered.

13.4.16 If after the third sonication, there is no noticeable reduction in solvent color, up to two additional aliquots of methylene chloride/acetone may be used. Record the total number of aliquots used if more than 3.

13.4.17 After the final sonication, decant the solvent then transfer all solids into the filter funnel and quantitatively rinse with methylene chloride.

13.5 The high concentration extraction is as follows:

13.5.1 Perform the following steps rapidly to avoid loss of more volatile compounds

13.5.2 Weigh 2 ±0.1 g of sample into a clean, unused 20 mL vial.

13.5.3 Add 2 g anhydrous sodium sulfate to sample and mix well until free-flowing.

13.5.4 Add 1.0 mL surrogate to samples, blanks and spikes.

13.5.5 For pesticides analysis, add 0.5 mL of pesticide spike solution and 8.5 mL hexane to matrix spikes and laboratory fortified blanks. For PCBs analysis, add 0.5 mL of PCB spike solution and 8.5 mL hexane. The total volume added must be 10.0 mL.

13.5.6 Sonicate using the 1/8 inch tapered microtip horn for two minutes at **OUTPUT CONTROL** setting 5 in **PULSE MODE** and **PERCENT-DUTY CYCLE** set at 50%.

13.5.7 Filter the extract through glass wool and collect in a pre-calibrated 15 mL vial. Assume a final volume of 10 mL even though not all of the extract will be recovered. Do NOT bring to volume in the pre-calibrated vial.

13.5.8 Cap the vial tightly and store at 4 ±2° C until cleanup and/or analysis.

13.5.9 If cleanup is required, proceed to Section 13.9.

13.6 Extraction of wipe samples is as follows:

13.6.1 Measure 1.0 mL of surrogate solution into the jar a wipe sample arrives in. Do not remove any sample material from the jar. For extraction blanks and blank spikes, use a 400 mL beaker containing 30.0 g of sodium sulfate.

13.6.2 Add 0.5 mL of pesticide or 0.5 mL of PCB spike solution to matrix and blank spikes, depending upon the analysis required.

13.6.3 Add approximately 50 mL of hexane to each jar. The wipe sample must be totally covered by the solvent.

13.6.4 Position the 3/4 inch horn in the sample jar approximately 1/2 inch below the solvent surface, above the wipe.

13.6.5 Sonicate for three minutes at full power (**OUTPUT CONTROL** at ten and **PERCENT-DUTY** cycle at 50%).

13.6.6 Repeat Sections 13.4.10 – 13.4.19, except use hexane instead of 1:1 methylene chloride/acetone and rinse with hexane instead of methylene chloride.

13.6.7 Repeat for each sample needing extracted.

13.7 Concentrate extracts as follows:

13.7.1 Add one or two clean boiling chips to each concentrator tube and attach a three-ball Snyder column.

13.7.2 Pre-wet the Snyder column by adding approximately 1 mL of methylene chloride through the top.

13.7.3 Place the concentrator in a water bath (80-90° C) where the concentrator tube is partially immersed and the entire lower rounded surface of the K-D flask is bathed with vapor.

13.7.4 At the proper rate of distillation, the glass column balls will actively chatter but not flood. Until chattering begins, rattle the Snyder column periodically.

13.7.5 Concentrate to an apparent volume of 3 mL. The concentration takes ten to fifteen minutes for low concentration extracts and five to ten minutes for high concentration extracts).

Note: When solvent volume is reduced below 1 mL using the K-D flask, semi-volatile analytes can be lost. If extracts are concentrated to dryness or near-dryness, the entire extraction must be repeated.

13.8 Perform the hexane solvent exchange as follows:

13.8.1 Remove and cool. The volume in the concentrator tube after cooling should be approximately 8 mL. Add 50 mL of hexane, wet the Snyder column and evaporate as before.

13.8.2 Transfer the extract to a 14 mL vial calibrated to 10 mL, rinsing the concentrator tube with several aliquots of hexane, totaling approximately 2 mL. Use only enough to bring the final volume to 10.0 mL. Repeat for each sample extracted.

13.9 Extract Cleanup

13.9.1 If necessary, pesticide extracts may be cleaned up using Florisil® columns (1000 mg or 20 g) as described in TriMatrix SOP GR-09-111.

13.9.2 All PCB extracts are cleaned up with sulfuric acid (TriMatrix SOP GR-09-110), and copper (TriMatrix SOP GR-09-109). If necessary, they are further cleaned up with Florisil® (TriMatrix SOP GR-09-111) or Florisil®/silica Gel (TriMatrix SOP GR-09-120).

13.10 Store finished extracts at 4 ±2° C in the GC refrigerator.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Extraction analysts are responsible for sample documentation and data integrity. All documentation must be correctly filled in. It is important to document extractions by correctly filling in, turning in and filing all paperwork accurately. This is mandatory for quality control and to provide clients with defensible data.

14.2 Analysts extracting a batch of up to 20 samples must input all extraction data to Element™.

14.3 Benchsheets and logbooks must be filled in completely to ensure that results are reported correctly and data is associated with the right quality control batch (Attachment 20.3).

14.4 If an internal chain-of-custody report is required, it is important the form be filled in accurately and completely.

14.5 All extraction laboratory hardcopy must be archived appropriately.

14.6 All extraction logbooks must be filled in completely and correctly (Attachment 20.4). Corrections must be made with one line through the error, dated and initials then the correction to the side. Do not perform a write-over or obliterate the error. "Z" out blank areas in logbooks.

15.0 QUALITY ASSURANCE

15.1 Extract an extraction blank (BLK) and blank spike (BS) daily or once per shift to demonstrate that interferences and contamination are under control. Extract a replicate blank spike when insufficient sample volume is received to perform a matrix spike duplicate.

15.2 Blank spikes are prepared by adding 0.5 mL of spike solution and 1.0 mL surrogate spike solution to 30 g of sodium sulfate. Always add 1.0 mL of surrogate solution to blanks.

15.3 An extraction batch is limited to no more than 20 samples.

15.4 When sufficient sample volume is received, extract a matrix spike (MS) and matrix spike duplicate (MSD) with every batch of up to 20 samples.

15.4.1 Prepare a matrix spike by measuring 0.5 mL of PCB spike solution or 0.5 mL of pesticide spike solution to 30 g of sample. Extract a matrix spike in accordance with every stop in the extraction procedure.

15.4.2 Always add 1.0 mL of surrogate to matrix spikes before extraction.

15.5 Sample matrix interference or laboratory-induced contamination can affect analyte recovery. Investigation and re-extraction may be necessary if surrogate and/or spiked compounds fail to pass laboratory established acceptance limits.

15.6 Perform corrective action for out-of-control quality control samples in accordance with TriMatrix SOP GR-04-101.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before preparation of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by extracting an initial demonstration of capability (IDC) study.

16.2 Prepare a spiking solution to give a final extract concentration mid-range in the GC calibration. Prepare independently from the calibration. Prepare four 30 g aliquots of sodium sulfate then measure spiking solution to each. Extract as normal samples in accordance with all extraction steps in the procedure and all subsequent cleanup steps.

16.3 After extraction and cleanup, have the four extracts analyzed by TriMatrix SOP GR-03-128 or GR-03-120. Whichever is appropriate to the spiked analyte.

16.4 Input results to the IDC spreadsheet (located on the laboratory intranet library) to calculate average recovery and relative standard deviation.

16.4.1 Recovery must be within laboratory established control limits and relative standard deviation must be less than or equal to 20%. If all analytes and criteria are acceptable, the IDC study is complete. The analyst is authorized to extract samples by this procedure.

16.4.2 If one or more analytes fail, the analyst must proceed according through the following steps.

16.5 If any analyte fails, locate and correct the source of error then repeat the study successfully for the failed analyte.

16.6 Repeated failure will indicate a problem with the procedure and/or analyst technique. Locate the problem and correct the procedure and/or techniques used then repeat the IDC study successfully.

16.7 Samples may not be prepared by the analyst until an IDC study has been successfully completed.

16.8 Repeat the demonstration of capability study annually as a continuing demonstration of capability (CDC) by one of the follow approaches:

16.8.1 Repeat the initial demonstration of capability study.

16.8.2 Use the last four results from an MDL study run exclusively by the analyst. Input to the IDC spreadsheet and submit as the CDC.

Note: Results might be abnormal or unacceptable because of the low concentration. If so, repeat the study with a higher concentration of spikes.

16.8.3 Successfully complete a blind performance testing sample extracted during the course of routine sample extraction. Extraction of the sample must have been done by the analyst.

16.8.4 Use four consecutively extracted blank spikes extracted during the course of routine sample preparation. Extraction must have been done by the analyst.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount required.

18.3 Follow all instructions in SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, February, 2007, Method 3550C, "Ultrasonic Extraction"

20.0 ATTACHMENTS

20.1 Standards Log Example

20.2 Disrupter Tuning Logbook Example

20.3 Preparation Batch Report Example

20.4 Extraction Logbook Example

Attachment 20.1
Standards Log Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
7030451

Description	Chlorinated Pesticide Spike	Expires	Sep-12-07
Standard Type	Analyte Spike	Prepared	Mar-12-07
Solvent	MeOH Lot#044685	Prepared By	Andrea S. Colborn
Final Volume (mls)	100	Department	Semivolatiles GC
Vials	1	Last Edit	Apr-20-07 09:54 by JLW

Analyte	CAS Number	Concentration	Units
4,4'-DDD	72-54-8	0.8	ug/mL
4,4'-DDD [2C]	72-54-8	0.8	ug/mL
4,4'-DDE	72-55-9	0.8	ug/mL
4,4'-DDE [2C]	72-55-9	0.8	ug/mL
4,4'-DDT	50-29-3	0.8	ug/mL
4,4'-DDT [2C]	50-29-3	0.8	ug/mL
Aldrin	309-00-2	0.8	ug/mL
Aldrin [2C]	309-00-2	0.8	ug/mL
alpha-BHC	319-84-6	0.8	ug/mL
alpha-BHC [2C]	319-84-6	0.8	ug/mL
alpha-Chlordane	5103-71-9	0.8	ug/mL
alpha-Chlordane [2C]	5103-71-9	0.8	ug/mL
beta-BHC	319-85-7	0.8	ug/mL
beta-BHC [2C]	319-85-7	0.8	ug/mL
delta-BHC	319-86-8	0.8	ug/mL
delta-BHC [2C]	319-86-8	0.8	ug/mL
Dieldrin	60-57-1	0.8	ug/mL
Dieldrin [2C]	60-57-1	0.8	ug/mL
Endosulfan I	959-98-8	0.8	ug/mL
Endosulfan I [2C]	959-98-8	0.8	ug/mL
Endosulfan II	33213-65-9	0.8	ug/mL
Endosulfan II [2C]	33213-65-9	0.8	ug/mL
Endosulfan Sulfate	1031-07-8	0.8	ug/mL
Endosulfan Sulfate [2C]	1031-07-8	0.8	ug/mL
Endrin	72-20-8	0.8	ug/mL
Endrin [2C]	72-20-8	0.8	ug/mL
Endrin Aldehyde	7421-93-4	0.8	ug/mL
Endrin Aldehyde [2C]	7421-93-4	0.8	ug/mL
Endrin Ketone	53494-70-5	0.8	ug/mL
Endrin Ketone [2C]	53494-70-5	0.8	ug/mL

Reviewed By _____ Date _____

Page 1 of 2

Analytical Standard Record
TriMatrix Laboratories, Inc.
7030451

gamma-BHC (Lindane)	58-89-9	0.8	ug/mL
gamma-BHC (Lindane) [2C]	58-89-9	0.8	ug/mL
gamma-Chlordane	5103-74-2	0.8	ug/mL
gamma-Chlordane [2C]	5103-74-2	0.8	ug/mL
Heptachlor	76-44-8	0.8	ug/mL
Heptachlor [2C]	76-44-8	0.8	ug/mL
Heptachlor Epoxide	1024-57-3	0.8	ug/mL
Heptachlor Epoxide [2C]	1024-57-3	0.8	ug/mL
Methoxychlor	72-43-5	0.8	ug/mL
Methoxychlor [2C]	72-43-5	0.8	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
A603424	(AMP) Organochlorine Pesticides	Feb-28-06	** Vendor **	Feb-29-08	Apr-20-07 09:54 by JLW	0.08

Reviewed By _____ Date _____

Page 2 of 2

Approved By: _____ 3-2-09
QA Officer

Approved By: _____ 3/2/09
Area Supervisor

Attachment 20.2
Disrupter Tuning Logbook Example

Date	Analyst	Fisher Scientific 550 Inst. #1 Final Tuner Reading	Pass / Fail (<20%)	Fisher Scientific 550 Inst. #2 Final Tuner Reading	Pass / Fail (<20%)
7/16/02	[illegible]	4%	Pass / Fail	5%	Pass / Fail
7/17/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/18/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/19/02	JEO	4%	Pass / Fail	8%	Pass / Fail
7/22/02	[illegible]	[illegible]	Pass / Fail	[illegible]	Pass / Fail
7/23/02	[illegible]	5%	Pass / Fail	9%	Pass / Fail
7/24/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/25/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/26/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/27/02	[illegible]	4%	Pass / Fail	8%	Pass / Fail
7/29/02	[illegible]	5%	Pass / Fail	8%	Pass / Fail
7/30/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/1/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/5/02	[illegible]	7%	Pass / Fail	5%	Pass / Fail
8/8/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/9/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/12/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/13/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/14/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/15/02	[illegible]	5%	Pass / Fail	8%	Pass / Fail
8/16/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/20/02	[illegible]	6%	Pass / Fail	8%	Pass / Fail
8/21/02	[illegible]	5%	Pass / Fail	8%	Pass / Fail
8/22/02	[illegible]	5%	Pass / Fail	8%	Pass / Fail
8/23/02	[illegible]	5%	Pass / Fail	9%	Pass / Fail
8/24/02	[illegible]	4%	Pass / Fail	8%	Pass / Fail
8/25/02	[illegible]	5%	Pass / Fail	7%	Pass / Fail
8/26/02	[illegible]	7%	Pass / Fail	8%	Pass / Fail
8/27/02	[illegible]	4%	Pass / Fail	8%	Pass / Fail
8/28/02	[illegible]	4%	Pass / Fail	8%	Pass / Fail

file: soniclog page: 16 of 50 revision: 1.0

Approved By: [signature] 3-2-09 QA Officer

Approved By: [signature] 3/2/09 Area Supervisor

Attachment 20.3
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0709022 Page 1 of 1

Printed: 9/21/2007 5:34:48PM

Semivolatiles GC, Soil, 3550B Sonication Extraction

Surrogate #1 = 7070018 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0708116	8081A PESTs (master list)	0708257	8081A PESTs (master list)	0708330	8081A PESTs (master list)
0708338	8081A PESTs (master list)				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (g)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0709022-BLK1		Aug-08-07 07:57	BJH	30	10	1000				BLANK	
0709022-BS1		Aug-08-07 07:57	BJH	30	10	1000		7030451	500	LCS	
0709022-BSD1		Aug-08-07 07:57	BJH	30	10	1000		7030451	500	LCS DUP	
0708116-01	C	Aug-08-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0709022-BLK2		Aug-15-07 07:57	BJH	30	10	1000				BLANK	
0709022-BS2		Aug-15-07 07:57	BJH	30	10	1000		7030451	500	LCS	
0708257-01	C	Aug-15-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0709022-BLK3		Aug-21-07 07:57	BJH	30	10	1000				BLANK	
0709022-BS3		Aug-21-07 07:57	BJH	30	10	1000		7030451	500	LCS	
0708330-04	A	Aug-21-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0708338-01	C	Aug-21-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0708338-02	C	Aug-21-07 07:57	BJH	30	10	1000				[redacted]	[redacted]

Comments:

Analyst Initials:

bch_TriMatrix.rpt

Approved By: _____ 3-2-09
QA Officer

Approved By: _____ BJH 3/2/09
Area Supervisor

Attachment 20.4
Extractions Logbook Example

TriMatrix Laboratories, Inc.

Organic Extraction Lab
Soil Extraction Logbook

Date: 3/11/04

Client	Project/ Submittal	Sample Number Range	LIMS Batch ID	Test or Parameter	Surrogate Spike Number and Volume		Matrix Spike Number and Volume	
EQ	36351-12	355751-756	212470	8082 PCB 3550	PLA.1-10	1.0mL	PLA.8-2	500mL
SAIC	36278-43	356038-046	213415	8330 EXP	PL3.12-2	500 mL	PLA1.2-13	1.9 mL
MDL				Full List Plus	PLA.2-7	100 mL	PL3.12-12 PL3.11-12	5.0mL 1mL
				PAK	3/11/04			

LIMS Sample ID	Bath Temp. °C	Initial Volume (g)	Final Volume (mL)	Analyst Initials
MPB111	90	30.0	10.0	MBS
355751		30.0		
355752		30.0		
355753		30.0		
355754		30.0		
355755		30.0		
355756		30.0		
MPB111	NA	2.0	10.0	DJM
356038		2.0		
356039		2.0		
356040		2.0		
356041		2.0		
356042		2.0		
356043		2.0		
356044		2.0		
356045		2.0		
356046		2.0		
356042MS		2.0		
356042MSD		2.0		
LFB111		2.0		
			PAK 3/11/04	

LIMS Sample ID	Bath Temp. °C	Initial Volume (g)	Final Volume (mL)	Analyst Initials
MDL 1	80	30.0	1.0	MBS
MDL 2		30.0		
MDL 3		30.0		
MDL 4		30.0		
MDL 5		30.0		
MDL 6		30.0		
MDL 7		30.0		
		PAK 3/11/04		

Reagent Information

CH_2Cl_2 Lot #: 030986
Hexane Lot #: 030702
Acetonitrile Lot #: Y30819
1:1 CH_2Cl_2/Acetone Reagent #: PLR1.48-7
Na_2SO_4 Reagent #: PLR1.51-2
Other #: NA

file: Prep Lab Soil Logbook page: 4 of 20 revision: 0.0

Approved By: PAK 3-2-09 QA Officer
Approved By: BJH 3/2/09 Area Supervisor

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Diesel Range Organics (DRO) Extraction

SW-846 Method 8015C
SW-846 Method 3550C

APPROVALS:

Area Supervisor: Andrea Colborn Date: 10/2/08
Andrea S. Colborn

QA Officer: Date: 10-2-08
Tom C. Boocher

Operations Manager: Date: 10/7/08
Jeff P. Glaser

Procedure Number: GR-09-123
Revision Number: 0.2

Date Initiated: 4/19/01
Effective Date: 10/15/08

Date Revised: 4/25/08
Pages Revised: All

By: Andrea S. Colborn

Total Number of Pages: 15

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the extraction of diesel range organics from soil, sediment, sludge or waste samples as diesel range organics (DRO).

1.2 The DRO corresponds to any component that elutes between decane (C_{10}) and octacosane (C_{28}) with an approximate boiling point range of 170 – 430° C.

1.3 This procedure corresponds to the low-concentration technique in the referenced extraction method.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update IV, Revision 3, February, 2007, Method 8015C, Nonhalogenated Organics by Gas Chromatography

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, February, 2007, Method 3550C, Ultrasonic Extraction

3.0 SUMMARY OF PROCEDURE

3.1 A 30 g sample aliquot is mixed with sodium sulfate to form a free-flowing powder then spiked with surrogate. The mixture is extracted with solvent three times, using ultrasonic extraction.

3.2 The combined extract is separated from the sample by filtration and/or centrifugation.

3.3 The extract is dried and concentrated to a final volume of 1.0 mL.

3.4 The concentrate is then ready for DRO analysis by gas chromatography (GC) using a flame ionization detector (FID).

4.0 PARAMETER OR COMPOUND LIST

4.1 Diesel Range Organics (DRO)

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration*, latest revision

5.2 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.3 TriMatrix SOP GR-03-101, *Semi-Volatiles Laboratory Quality Control Corrective Actions*, latest revision

5.4 TriMatrix SOP GR-10-125, *Method Detection Limit*, latest revision

5.5 TriMatrix SOP GR-03-122, *Diesel Range Organics (DRO)*, latest revision

5.6 TriMatrix SOP GR-10-113, *Laboratory Balance Calibration and Verification*, latest revision

5.7 TriMatrix SOP GR-09-128, *Mixing and Grinding Samples for Organic Extractions*, latest revision

5.8 TriMatrix SOP GR-09-103, *Extraction of BNA Semi-Volatiles from Soil, Sediment and Sludge*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Organic compounds including animal or vegetable oil/grease, chlorinated hydrocarbons, phenols, phthalate esters and motor oils can have components that elute within the DRO window. This is addressed in TriMatrix SOP GR-03-122.

6.2 Interferences can be from contaminants in solvents, reagents, glassware and other sample processing equipment.

6.2.1 Rinse washed glassware with methylene chloride just prior to use.

6.2.2 Use only pesticide grade (or better) solvents.

6.2.3 Rinse sodium sulfate with methylene chloride before use.

6.2.4 Extraction blanks must be extracted with samples. Refer to Section 15.0.

6.3 Matrix and/or laboratory-induced interferences can affect analyte and/or surrogate spike concentrations. Sample re-extraction may be necessary if quality control criteria are exceeded.

6.4 Use only PTFE dispensing bottles for solvent rinsing. Do not use plastic equipment for this procedure.

7.0 SAFETY PRECAUTIONS

7.1 Wear a laboratory coat and approved safety glasses when in the extractions laboratory. Wear disposable gloves whenever samples or reagents are handled.

7.2 Follow all safety instructions as outlined in the TriMatrix laboratory safety manual and chemical hygiene plan.

7.3 For proper spill response and waste disposal, refer to TriMatrix GR-15-102.

7.4 All chemicals in the laboratory must be treated as a potential health hazard. Reduce exposure to the lowest possible level.

7.5 Material safety data sheets (MSDS) are available on the laboratory intranet library from the extraction laboratory office computer.

7.6 NO solvents or solvent work is to be done in the extraction laboratory office. Chemicals are not permitted in laboratory offices.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Collect samples in screw-cap glass jars (60 mL, 125 mL or 250 mL) with PTFE-lined lids.

8.2 Handle sample containers with care to avoid breakage.

8.3 Store samples in the walk-in cooler at 4 ±2° C when not in use.

8.4 Extract samples within 14 days of the collection date. Extracts must be analyzed within 40 days of the extraction date.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Heavy duty Pyrex beakers, 400 mL and 600 mL

9.2 500 mL Kuderna-Danish evaporation flasks

9.3 10 mL graduated concentrator tubes

9.4 Snyder columns, 3-ball macro and 2-ball micro

9.5 Variable temperature steam bath in the fume hood

9.6 Analytical balance, capable of accurately weighing to the nearest 0.1 mg

9.7 Filter paper, qualitative, Fisher P8, VWR 415 or Whatman 41

9.8 Tongue depressors, wooden

9.9 Disposable glass Pasteur pipettes, 2 mL

9.10 Methylene chloride-rinsed boiling chips, approximately 10/40 mesh (PTFE) or equivalent

9.11 2, 10, 15, and 40 mL vials with PTFE-lined screw cap lids

9.12 Hamilton microsyringes, gastight, 25, 100, 500, 1000 µL

9.13 Filter funnels for drying extracts before concentration, glass

9.14 Erlenmeyer flasks, 250 and 500 mL

9.15 Top-loading balance, capable of accurately weighing to the nearest 0.1 g

9.16 Ultrasonic dismembrators, 2 units, with 3/4 inch horn

9.17 Sonabox for each dismembrator unit

9.18 Centrifuge, for extracts with fine particles that pass through the filter paper

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Tune the ultrasonic dismembrator at the beginning of each work shift.

10.2 Clean the disrupter horn before each sample.

10.3 Perform a thorough cleaning of all equipment at the end of the work shift.

10.4 Clean glassware in accordance with TriMatrix SOP GR-16-100.

11.0 CHEMICALS AND REAGENTS

11.1 Sodium sulfate (granular, anhydrous), Na_2SO_4, ACS

Note: Extract a blank for each lot of sodium sulfate before use to demonstrate that there is no interference above the method detection limit (MDL) from the sodium sulfate. If interferences are detected, place in a muffle furnace at 400° C for 4 hours and retest successfully before using.

11.2 Methanol, pesticides grade or better

11.3 Methylene chloride, pesticides grade or better

11.4 o-Terphenyl, certified purity greater than 95%

11.5 Laboratory reagent water, Milli-Q system, pre-extracted with extraction solvent before use

12.0 STANDARDS PREPARATION

12.1 Input all spiking standards data into the laboratory information management system (Element™) as individual solutions.

12.2 The expiration of working dilutions is 6 months from the date prepared if stored without headspace at 4 ±2°C.

12.3 The expiration of stock solutions prepared from neat materials or purchased as certified standards is 1 year from the date prepared or opened if stored without headspace at 4 ±2°C.

12.4 A prepared standard expiration date is not to exceed any manufacturer's expiration date.

12.5 Dispose of expired standards appropriately.

12.6 Monitor the inventory and expiration date of all stock standards to avoid expedited shipping fees.

12.7 Store surrogate/spiking standards in 40 mL amber vials labeled with the following information:

12.7.1 Standard name

12.7.2 Identification number

12.7.3 Date prepared

12.7.4 Analyst initials

12.7.5 Solvent

12.7.6 Concentration with units

12.7.7 Expiration date

12.8 Store all surrogate/spiking standards in the extractions laboratory refrigerator at 4 ±2° C. Remake any standard solution containing less than 5 mL, that shows evidence of evaporation or degradation, or that fails quality control limits.

12.9 Dispose of standard solutions appropriately.

12.10 All standard preparation calculations are verified and documented by Element™. Refer to Attachment 20.2.

12.11 All prepared standards must be verified and documented by analysis before using.

12.11.1 Prepare a dilution of the standard at the same concentration to be used.

12.11.2 Give to the analysis laboratory.

12.11.3 Review results after analysis to verify the concentration using default 80-120% acceptance limits. Results should be well within the acceptance limits. If not acceptable or if the analyst suspects a problem exists, remake the standard successfully and repeat the analysis check.

12.11.4 If results indicate the standard was prepared correctly and accurately, it may be used for sample extractions and/or analysis.

12.12 Diesel fuel #2 is purchased as a certified 50,000 ug/mL stock standard in methylene chloride for quality control spiking. This standard is not used for instrument calibration.

12.13 Prepare a 2,000 ug/mL DRO blank spike (BS) and matrix spike (MS) spiking solution in methylene chloride from the 50,000 ug/mL stock standard as follows:

$$\frac{\text{50000 µg \#2 diesel fuel}}{\text{mL}} \Big| \frac{\text{2 mL}}{\text{50 mL}} = \text{2000 ug/mL \#2 diesel}$$

12.14 Prepare a 200 ug/mL o-terphenyl surrogate spiking standard by serial dilution in methanol as follows:

12.14.1 Use the analytical balance located in the instrumental laboratory. Make sure the balance has been calibrated that day in accordance with TriMatrix GR-10-113.

12.14.2 Tare a 25 mL volumetric flask on the balance. Do not add solvent.

12.14.3 Weigh and record 0.5000 g of o-terphenyl into the flask to the nearest 0.0001 g.

12.14.4 Once the o-terphenyl is weighed and recorded, add approximately 20 mL of methanol then swirl and check for dissolution. If the o-terphenyl does not dissolve, do not dilute to volume. Continue to swirl until dissolved or if dissolution does not appear to be happening, perform the following:

12.14.4.1 Stopper and position in an ultrasonic bath to aid in dissolution.

12.14.4.2 Closely monitor the solution during sonication as the ultrasonic bath will warm the standard. Do not allow the solution to get warm.

12.14.4.3 Remove from the bath as soon as the o-terphenyl begins to dissolve.

12.14.4.4 Swirl until dissolved.

12.14.4.5 After dissolving, dilute to volume with methanol. Invert and shake vigorously several times to mix thoroughly. Concentration is 20,000 ug/mL.

12.14.5 Prepare the 200 ug/mL solution as follows:

12.14.5.1 Fill a 100 mL volumetric flask with about 80 mL of methanol.

12.14.5.2 Measure and add 1.0 mL of the 20,000 ug/mL solution into the flask.

12.14.5.3 After diluting to volume with methanol, invert and shake vigorously several times to mix thoroughly. Concentration is 200 ug/mL.

12.14.5.4 Pour into 5 pre-labeled 16 mL vials and store without headspace at 4 ±2° C until needed. Discard any residual solution. Expiration is 6 months from the date prepared.

12.14.6 Pour the remaining 20,000 ug/mL solution into a pre-labeled 20 mL vial and store without headspace at 4 ±2° C until needed. Expiration is 12 months from the date prepared.

13.0 ANALYTICAL PROCEDURE

13.1 Tune the disruptor horns at the beginning of each work shift. Refer to the instrument manual for tuning instructions.

13.2 Perform the sample extraction as follows:

13.2.1 Remove and discard any extraneous foreign objects such as sticks, leaves or rocks.

13.2.2 After removing, homogenize the sample in accordance with TriMatrix SOP GR-09-128.

13.2.3 Label all glassware with the complete sample number and extraction type. For example, label whether the extraction is to be a matrix spike (MS), matrix spike duplicate (MSD) or blank spike (BS/LFB).

13.2.4 Perform the following steps rapidly to minimize the evaporation of volatile compounds.

13.2.4.1 For sandy to dry matrices, weigh 30 g of sample into a 400 mL beaker and record to the nearest 0.1 g (example: 30.2 g). Then, add 20-25 g of anhydrous sodium sulfate. Mix well. The mixture should have a sandy appearance and be free-flowing after mixing. If not, mix in additional sodium sulfate until it is.

13.2.4.2 For wet to gummy matrices, weigh 30 g of sample into a 400 mL beaker and record to the nearest 0.1 g (example: 30.2 g). Then, add approximately 60 g of anhydrous sodium sulfate. Mix well. The mixture should have a sandy appearance and be free-flowing after mixing.

13.2.4.3 If not free-flowing, mix in additional sodium sulfate until it is. Record the total amount used to match with quality control samples.

13.2.4.4 Add 200 µL of 200 ug/mL surrogate (Section 12.14) to all extractions including extraction blanks (BLK), blank spikes (BS/LFB) and matrix spikes (MS/MSD). Concentration in the final extract volume and sample is as follows:

$$\frac{\frac{200\ \mu g\ \text{o-terphenyl}}{mL}}{\frac{0.2\ mL}{1\ mL\ \text{extract}}} = 40\ \text{ug/mL o-terphenyl (in extract)}\ (1.3_3\ \text{mg/kg in 30 g sample})$$

13.2.4.5 Add 500 µL of the 2,000 ug/mL spiking standard (Section 12.13) to blank and matrix spikes.

13.2.4.6 After spiking, immediately add 100 mL of methylene chloride.

13.2.5 Position the bottom surface of the 3/4 inch disrupter horn tip about 1/2 inch below the solvent surface but above the sediment layer.

13.2.6 Extract ultrasonically for 3 minutes at full power (output control knob set at 10 and percent-duty cycle knob set at 50 percent).

13.2.7 Have a K-D concentrator set up to receive the extraction solvent as follows:

13.2.7.1 Insert a 100 mm filter funnel lined with qualitative filter paper.

13.2.7.2 Add approximately 1 inch of anhydrous sodium sulfate to the filter funnel.

13.2.7.3 Rinse the filter paper and sodium sulfate with methylene chloride.

13.2.7.4 Insert into the top of the K-D concentrator flask with attached concentrator tube.

13.2.8 Decant the extraction solvent into the filter funnel and let drain to the K-D flask.

13.2.9 Repeat the extraction two more times with two additional 100 mL volumes of methylene chloride. Decant to the K-D flask after each extraction. On the final extraction, pour all sediment from the extraction beaker into the filter funnel and rinse with methylene chloride. Collect all rinsings in the K-D flask.

13.2.10 Concentrate extracts as follows:

13.2.10.1 Add one or two clean boiling chips to the K-D flask and attach a three-ball Snyder column.

13.2.10.2 Pre-wet the Snyder column by adding approximately 1 mL methylene chloride to the top.

13.2.10.3 Position in the water bath (50-60° C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor.

13.2.10.4 At the proper rate of distillation, the column will actively chatter but the chambers will not flood with condensed solvent. Adjust the depth of the concentrator tube accordingly.

13.2.10.5 When the apparent volume of liquid reaches 1 mL (usually within 10 to 15 minutes), remove the K-D apparatus from the bath. Let drain and cool for at least 10 minutes before removing the Snyder column.

Note: Do not let the concentration go to dryness. If it does, repeat the entire extraction.

13.3 After cooling, remove the Snyder column and rinse the lower joints of the column and the flask into the concentrator tube with 1-2 mL of methylene chloride.

13.4 Add another clean boiling chip to the concentrator tube and attach a 2-ball micro-Snyder column. Pre-wet the column with 0.5 mL methylene chloride then partially immerse in the 60-70° C water bath. Do not immerse too deep or bumping of the extract and/or flooding of the micro-Snyder column will occur. Immerse only to the point that the balls in the column actively chatter but do not flood.

13.5 When the apparent volume reaches 0.5 mL, remove from the water bath and allow to cool for 10 minutes (do not remove the micro-Snyder column before the cool-down).

Note: Do not let the concentration go to dryness. If it does, repeat the entire extraction.

13.6 After cooling, remove the column and transfer the extract to a pre-calibrated 1 mL vial using a clean, disposable, Pasteur pipette.

13.7 Rinse the lower joints of the micro-Snyder column with approximately 0.2 mL of methylene chloride into the concentrator tube then transfer the rinse to the 1 mL vial also.

13.8 Adjust the final volume in the vial to 1 mL with methylene chloride then cap tightly.

13.9 Store extracts at 4 ±2° C in the GC refrigerator until analysis.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Analysts are responsible for data quality and for correctly filling in all paperwork, laboratory notebooks and other documentation. This is required for quality assurance and to provide the client with fully traceable and defensible data.

14.2 Input all required extraction data to the laboratory information management system (LIMS/Element™).

14.3 Batch reports must be filled in completely to insure that results are reported correctly and data is associated with the batch quality control.

14.4 If internal chain-of-custody (CoC) is required, it is important that the CoC form be filled in completely and correctly.

14.5 Logbooks must be filled in completely and correctly. **Corrections are to be made with a lineout, initials, and date, not a write-over**. Blank lines in the logbook must be Z'd out.

15.0 QUALITY ASSURANCE

15.1 Extract an extraction blank (BLK), blank spike (BS/LFB), matrix spike (MS) and matrix spike duplicate (MSD) for each extraction batch of up to 20 samples or each day samples are extracted. Whichever is more frequent.

15.2 Add 200 µL of 200 ug/mL surrogate (Section 12.14) to all extractions including extraction blanks (BLK), blank spikes (BS/LFB) and matrix spikes (MS/MSD).

15.3 Prepare the extraction blank by extracting 30 g of sodium sulfate as a sample. Add extra sodium sulfate to match the maximum amount of sodium sulfate used in samples. Extract and evaluate as a 30 g sample.

15.4 Prepare the blank spike by adding 500 µL of 2000 ug/mL DRO spiking solution (Section 12.13) to 30 g of sodium sulfate. Add extra sodium sulfate to match the maximum amount of sodium sulfate used in samples. Extract and evaluate as a 30 g sample with a 33.3_3 mg/kg spike.

$$\frac{\text{2000 µg \#2 diesel fuel}}{\text{mL}} \Big| \frac{\text{0.5 mL}}{\text{30 g}} = 33.3_3 \text{ mg/kg \#2 diesel}$$

15.5 Prepare matrix spikes by adding 500 µL of 2000 ug/mL DRO spiking solution (Section 12.13) to 30 g of sample before extracting. Add the same amount of sodium sulfate to match the amount of sodium sulfate used in the unspiked sample. Extract and evaluate as a 30 g sample with a 33.3_3 mg/kg spike.

15.6 An out-of-control blank spike requires the generation of a non-conformance report to initiate root-cause analysis and corrective action. These need coordinated with the analysis laboratory and with the quality assurance department.

15.7 Address all quality control issues in accordance with TriMatrix SOP GR-03-101.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a successful Initial Demonstration of Capability (IDC) study.

16.2 Perform the IDC as follows:

16.2.1 Prepare a 2,000 ug/mL DRO blank spiking standard from a source other than that used for calibration of the DRO gas chromatograph (GC) instrument.

16.2.2 Set up four 30 g sodium sulfate blank spikes, spike with 0.5 mL of the spiking standard (and surrogate), and extract as samples.

16.2.3 Analyze in accordance with TriMatrix SOP GR-03-122 then input results to the IDC spreadsheet located on the laboratory intranet library. Average percent recovery must fall within LIMS blank spike acceptance limits. Relative standard deviation must be ≤20%.

16.3 If either criterion is not met, locate and correct the source of the problem and repeat the study successfully.

16.4 Repeated IDC failure will indicate a problem with the procedure and/or techniques used. If this occurs, locate and correct the source of the problem, revise the procedure and/or techniques used and repeat the study successfully.

16.5 Samples may not be extracted by the analyst until an IDC study has been successfully completed.

16.6 Copies of successful IDC studies (Spreadsheet and extraction data) must be submitted to the quality assurance department for training documentation.

16.7 A Continuing Demonstration of Capability (CDC) study is required annually. The CDC can be accomplished by any of the following approaches:

16.7.1 By repeating the IDC study.

16.7.2 By using the last four results from the annual method detection limits (MDL) study if extracted exclusively by the analyst.

16.7.3 By inputting four consecutive blank spike results obtained during the course of routine sample processing to the IDC spreadsheet if extracted exclusively by the analyst.

16.7.4 By exclusively and successfully extracting a blind PT study sample during the course of routine sample processing.

16.8 A Method Detection Limit (MDL) study must be extracted annually in accordance with TriMatrix GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update IV, Revision 3, February, 2007, Method 8015C, Nonhalogenated Organics by Gas Chromatography

19.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, February, 2007, Method 3550C, Ultrasonic Extraction

20.0 ATTACHMENTS

20.1 Preparation Batch Report Example

20.2 Standards Log Example

Attachment 20.1
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0804647 Page 1 of 1

Printed: 6/16/2008 1:45:36PM

Semivolatiles GC, Soil, 3550B Sonication Extraction

Batch comments:

no bsd or ms/msd per work order comments
DO NOT ADD ANY SAMPLES TO THIS BATCH

				Initial	Final	..... Client			
Lab Number	*Container*	*Prepared*	*By*	*(g)*	*(mL)*	*Source ID*	*Spike ID*	*uL Spike*	*Comments*
0804444-04	A	Apr-25-08 16:15	ASC	30	1	[redacted]			
DRO EPA 8015B									
0804647-BLK1		Apr-25-08 16:47	ASC	30	1				same as batch 0804646
0804647-BS1		Apr-25-08 16:47	ASC	30	1		7120133	500	same as batch 0804646

Comments:	Analyst Initials:

bch_TM_byAnalysis.rpt

Approved By: QA 10-2-08 **QA Officer**

Approved By: ASC 10/2/08 **Area Supervisor**

Attachment 20.2
Standards Log Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

7030944

Description:	(AMP) Diesel Fuel #2 2o	Expires:	Apr-23-08
Standard Type:	Other	Prepared:	Aug-31-06
Solvent:	Solvent Lot #A046397	Prepared By:	** Vendor **
Final Volume (mls):	1	Department:	Expired
Vials:	1	Last Edit:	May-13-08 13:05 by JLW

Restek part#31259
Lot: A046397
Received 3/27/07

Analyte	CAS Number	Concentration	Units
DRO - 8015B (C10-C28)		50000	ug/mL

Approved By: [signature] 10-2-08
QA Officer

Approved By: ASC 10/2/08
Area Supervisor

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Separatory Funnel Liquid-Liquid Extraction of Diesel Range Organics (DRO)

EPA Method 8015C
EPA Method 3510C

APPROVALS:

Area Supervisor: Andrea Colborn Date: 9/9/08
Andrea S. Colborn

QA Officer: _____ Date: 9-5-08
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 9/5/08
Jeff P. Glaser

Procedure Number: GR-09-124
Revision Number: 0.1

Date Initiated: 4/19/01
Effective Date: 9/10/08

Date Revised: 8/15/08
Pages Revised: All

By: Andrea S. Colborn
Total Number of Pages: 17

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the extraction of diesel range organics (DRO) from aqueous samples and for extract concentration in preparation for gas chromatographic analysis.

1.2 The DRO corresponds to an alkane range of $C_{10} - C_{28}$ and a boiling point range of 170 – 430° C.

1.3 This procedure is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results before extracting samples.

1.4 This procedure may also be applicable to fuel types and petroleum hydrocarbons other than DRO if the following is adhered to.

1.4.1 The fuel type or petroleum fraction must be clearly defined as a petroleum industry standard.

1.4.2 To be used for fuel types or petroleum hydrocarbons other than DRO, a demonstration that gas chromatographic conditions, including the GC column are appropriate for the analysis.

1.4.3 The analyst must also perform an initial demonstration of proficiency (IDC) study for each fuel type or petroleum hydrocarbon before samples may be analyzed.

1.4.4 The boiling point range or carbon number range of the material must be carefully defined.

1.4.5 The quantitation approach must be optimized to match the boiling point range or carbon number range chosen.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update IV, Revision 3, February, 2007, Method 8015C, Nonhalogenated Organics by Gas Chromatography

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision 3, December, 1996, Method 3510C, "Separatory Funnel Liquid-Liquid Extraction".

3.0 SUMMARY OF PROCEDURE

3.1 A measured volume of sample (nominally, 1 L) is spiked with surrogate and serially extracted with methylene chloride.

3.2 The extract is chemically dried and concentrated to a volume of 1.0 mL in preparation for analysis.

4.0 PARAMETER OR COMPOUND LIST

4.1 Diesel Range Organics (DRO) - defined as any peak eluting between the lower retention time limit of C_{10} and the upper retention time limit of C_{28} n-alkanes.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration*, latest revision

5.2 TriMatrix SOP GR-16-100, *Glassware Cleaning and Prep for Organics,* latest revision

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal,* latest revision

5.4 TriMatrix SOP GR-03-122, *Diesel Range Organics (DRO)*, latest revision

5.5 TriMatrix SOP GR-10-101, *Semi-Volatiles Laboratory Quality Control Corrective Actions*, latest revision

5.6 TriMatrix SOP GR-09-123, *Diesel Range Organics (DRO) Extraction (soils)*, latest revision

5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Other organic compounds including animal and vegetable oils, grease, chlorinated hydrocarbons, phenols and phthalate esters are measurable as DRO if meeting the definition of Section 4.1.

6.2 Use only reagents and solvents that do not interfere in the analysis.

6.3 Clean glassware in accordance with TriMatrix SOP GR-16-100.

6.4 Sample matrix interference or from laboratory-induced contamination can affect recovery of analytes and spiked compounds. Re-extraction of samples might be necessary if surrogate or spiked compounds fail quality control acceptance limits.

7.0 SAFETY PRECAUTIONS

7.1 Wear a laboratory coat and approved safety glasses when in the extractions laboratory. Wear disposable gloves whenever samples or reagents are handled.

7.2 Follow all safety instructions as outlined in the TriMatrix laboratory safety manual and chemical hygiene plan.

7.3 For proper spill response and waste disposal, refer to TriMatrix GR-15-102.

7.4 All chemicals in the laboratory must be treated as a potential health hazard. Reduce exposure to the lowest possible level.

7.5 Material safety data sheets (MSDS) are available on the laboratory intranet library from the extraction laboratory office computer.

7.6 No solvents or solvent work is to be in the extraction laboratory office. Chemicals are not permitted in laboratory offices where food is eaten. No food is to be stored in laboratory offices although it may be eaten there.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Collect samples in a one liter amber glass container. Store at 4 ±2° C from the time of collection.

8.2 Extract within 7 days of the collection date/time and analyze within 40 days of extracting.

8.3 When not in use, store samples in the walk-in cooler.

8.4 Use care when handling samples and glassware to avoid breakage.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Evaporation flask, Kuderna-Danish, 500 mL, attach the concentrator tube with a blue clip

9.2 Graduated concentrator tubes, 10 mL

9.3 Snyder columns, 3-ball macro and 2-ball micro

9.4 Variable temperature steam bath in the fume hood

9.5 Analytical balance, capable of accurately weighing to the nearest 0.1 mg

9.6 Filter paper, qualitative, Fisher P8, VWR 415 or Whatman 41

9.7 Disposable glass Pasteur pipettes, 2 mL

9.8 Methylene chloride-rinsed boiling chips, approximately 10/40 mesh (PTFE) or equivalent

9.9 Vials with PTFE-lined screw cap lids, 2, 10, 15, 40 mL

9.10 Microsyringes, gastight, 25, 100, 500, 1000 µL

9.11 Filter funnels for drying extracts before concentration, glass

9.12 Erlenmeyer flasks, 250 mL

9.13 Separatory Funnel: 2000 mL with polytetrafluoroethylene (PTFE) stopcock and stopper

9.14 Concentrator Tube, Kuderna-Danish: 10 mL graduated (Kontes K-570050-1025 or equivalent).

9.15 Glass autosampler vials with PTFE-lined screw-caps, calibrated to 1.0 mL in accordance with TriMatrix SOP GR-09-106

9.16 Amber vials with PTFE-lined screw-caps, 40 mL

9.17 Graduated cylinder, glass, 1000 mL, type B

9.18 Volumetric flasks, 50, 100 and 1000 mL, type A

9.19 Centrifuge, for breaking up emulsions

9.20 Phase separation paper

9.21 Glass wool

9.22 pH indicator strips: pH range 0-14

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 If a sample discolors or deposits petroleum residue on extraction or concentration glassware, thoroughly rinse the glassware with methylene chloride prior to washing in accordance with TriMatrix SOP GR-16-100.

10.2 Replace and/or repair chipped or broken glass as needed.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water is pre-extracted with methylene chloride to minimize interferences as follows:

11.1.2 Add 1000 mL of laboratory reagent water from the MilliQ system to a 1000 mL separatory funnel.

11.1.2 Add 60 mL of methylene chloride and extract by vigorously shaking for at least 2 minutes with frequent venting.

11.1.2 Let the methylene chloride settle for at least 10 minutes then drain and discard appropriately.

11.1.2 The remaining mixture is then ready for use as an extraction blank or blank spike.

Note: Rinse each separatory funnel used for sample extractions with methylene chloride just prior to use.

11.2 Methylene chloride, methanol and acetone, pesticide grade or better

11.3 Sodium sulfate (granular, anhydrous Na_2SO_4), ACS

Note: Sodium sulfate may be used without solvent rinsing or baking in the muffle furnace if the extraction blank is below the method detection limit for all target analytes.

Note: Extract a blank for each lot of sodium sulfate before use to demonstrate that there are no interferences from the sodium sulfate at or above the method detection limit (not reporting limit). If a tested lot indicates contamination, bake at 400° C for 4 hours and retest. If acceptable, it may then be used

11.4 Sulfuric acid solution (1:1 v/v), carefully add 500 mL concentrated sulfuric acid to 500 mL of laboratory reagent water (not pre-extracted with solvent) in a glass container.

Note: This solution generates significant heat. Add acid slowly to the water and cool before using. Never add water to acid.

12.0 STANDARDS PREPARATION

12.1 Input all spiking standards data into the laboratory information management system (Element™) as individual solutions.

12.2 The expiration of working dilutions is 6 months from the date prepared if stored without headspace at 4 ±2° C.

12.3 The expiration of stock solutions prepared from neat materials or purchased as certified standards is one year from the date prepared or opened if stored without headspace at 4 ±2° C.

12.4 A prepared standard expiration date is not to exceed any manufacturer's expiration date.

12.5 Dispose of expired standards appropriately.

12.6 Monitor the inventory and expiration date of all stock standards to avoid expedited shipping fees.

12.7 Store surrogate/spiking standards in 40 mL amber vials labeled with the following information:

12.7.1 Standard name

12.7.2 Identification number

12.7.3 Date prepared

12.7.4 Analyst initials

12.7.5 Solvent

12.7.6 Concentration with units

12.7.7 Expiration date

12.8 Store all surrogate/spiking standards in the extractions laboratory refrigerator at 4 ±2° C. Remake any standard solution containing less than 5 mL, that shows evidence of evaporation or degradation, or that fails quality control limits.

12.9 Dispose of standard solutions appropriately.

12.10 All standard preparation calculations are verified and documented by Element™.

12.11 All prepared standards must be verified and documented by analysis before using.

12.11.1 Prepare a dilution of the standard at the same concentration to be used.

12.11.2 Give to the analysis laboratory.

12.11.3 Review results after analysis to verify the concentration using default 80-120% acceptance limits. Results should be well within the acceptance limits. If not acceptable or if the analyst determines a problem exists, remake the standard successfully and repeat the analysis check.

12.11.4 If results indicate the standard was prepared correctly and accurately, it may be used for sample extractions and/or analysis.

12.12 Diesel fuel #2 is purchased as a certified 50,000 ug/mL stock standard in methylene chloride for quality control spiking. This standard is not used for instrument calibration.

12.13 Prepare a 2,000 ug/mL DRO blank spike (BS) and matrix spike (MS) spiking solution in methylene chloride from the 50,000 ug/mL stock standard as follows:

$$\frac{\frac{50000\ \mu g\ \#2\ diesel\ fuel}{mL} \mid \frac{2.0\ mL}{50\ mL}}{} = 2000\ ug/mL\ \#2\ diesel$$

12.14 Prepare a 200 ug/mL o-terphenyl surrogate spiking standard by serial dilution in methanol as follows:

12.14.1 Use the analytical balance located in the instrumental laboratory. Make sure the balance has been calibrated that day in accordance with TriMatrix GR-10-113.

12.14.2 Tare a 25 mL volumetric flask on the balance. Do not add solvent.

12.14.3 Weigh and record 0.5000 g of o-terphenyl into the flask to the nearest 0.0001 g.

12.14.4 Once the o-terphenyl is weighed and recorded, add approximately 20 mL of methanol then swirl and check for dissolution. If the o-terphenyl does not dissolve, do NOT dilute to volume. Continue to swirl until dissolved or if dissolution does not appear to be happening, perform the following:

12.14.4.1 Stopper and position in an ultrasonic bath to aid in dissolution.

12.14.4.2 Closely monitor the solution during sonication as the ultrasonic bath will warm the standard. Do not allow the solution to get warm.

12.14.4.3 Remove from the bath as soon as the o-terphenyl begins to dissolve.

12.14.4.4 Swirl until dissolved.

12.14.4.5 After dissolving, dilute to volume with methanol. Invert and shake vigorously several times to mix thoroughly. Concentration is 20,000 ug/mL.

12.14.5 Prepare the 200 ug/mL solution as follows:

12.14.5.1 Fill a 100 mL volumetric flask with about 80 mL of methanol.

12.14.5.2 Measure and add 1.0 mL of the 20,000 ug/mL solution into the flask.

12.14.5.3 After diluting to volume with methanol, invert and shake vigorously several times to mix thoroughly. Concentration is 200 ug/mL.

12.14.5.4 Pour into 5 pre-labeled 16 mL vials and store without headspace at 4 ±2° C until needed. Discard any residual solution. Expiration is 6 months from the date prepared.

12.14.6 Pour the remaining 20,000 ug/mL solution into a pre-labeled 20 mL vial and store without headspace at 4 ±2° C until needed. Expiration is 12 months from the date prepared.

13.0 ANALYTICAL PROCEDURE

13.1 Assemble glassware and samples.

13.1.1 Rinse all glassware with methylene chloride prior to use.

13.1.2 Label each separatory funnel, Erlenmeyer flask, K-D concentrator and concentrator tube with the following:

13.1.2.1 Entire sample number

13.1.2.2 Analysis

13.1.2.3 "S" for surrogate addition (Erlenmeyer flasks only)

13.1.2.4 "L" for spike addition, where applicable (Erlenmeyer flasks only)

13.1.2.5 Final extract volume, in mL

13.2 Determine sample volumes as follows:

13.2.1 Shake the sample bottle by inverting it at least three or four times.

13.2.2 If sample volume received is >500 mL, proceed with the extraction.

13.2.3 If sample volume received is <500 mL, consult the project chemist to see if the client wishes to proceed since reporting limits will be elevated with the smaller volume.

13.2.4 If a sample is received with a significant amount of sediment, consult the project chemist to determine how the client requires the sediment addressed. Options are as follows:

13.2.4.1 Let the sediment settle and extract only the water phase.

13.2.4.2 Extract only the sediment phase.

13.2.4.3 Extract both phases separately.

Note: Do not confuse sediment with "dirty water" samples containing particulate material. Such particulates are inherently part of the sample and need included in the water extraction.

13.3 Check and note the initial pH. If necessary, adjust pH to <2.0 using 2.0 mL aliquots 1:1 (v/v) sulfuric acid.

13.4 Add 200 µL of 200 ug/mL ortho-terphenyl surrogate standard to all samples, extraction blanks (BLK), blank spikes (BS/LFB) and matrix spikes (MS/MSD).

13.5 For each extraction batch of up to 20 samples, prepare a matrix spike (MS) and matrix spike duplicate (MS) by spiking with 500 µL of the 2000 ug/mL DRO spiking solution prior to extracting.

13.6 Extract a blank and blank spike for each batch of up to 20 samples. At least one extraction blank and blank spike must be extracted for each day samples are extracted.

13.7 Prior to extraction, add 100 mL of methylene chloride to the sample bottle to rinse the inside. Cap and shake the bottle with the solvent then transfer to the sample's separatory funnel. Extract the sample by shaking for two minutes with frequent venting.

13.8 After shaking, let each phase separate for a minimum of 10 minutes then drain the bottom phase into a 250 mL Erlenmeyer flask. If an emulsion forms in the separatory funnel, it can be broken up as follows:

13.8.1 Place the separatory funnel on the holding rack and let the sample settle for 15-30 minutes.

13.8.2 Rattle the funnel periodically to begin breaking up the emulsion.

13.8.3 Drain the entire emulsion/methylene chloride mix into a methylene chloride rinsed 125 mL separatory funnel and add 20 mL of methylene chloride then shake.

13.8.4 If separation occurs after vigorously shaking, drain the methylene chloride into a 250 mL Erlenmeyer flask and transfer the water phase back to the 2 L separatory funnel, completing the transfer by rinsing the funnel methylene chloride.

13.8.5 If separation does not occur, drain the emulsion into PTFE-lined centrifuge tubes. Centrifuge for 5 minutes at 4500 rpm. Once centrifuged, drain the water layer (including any heavy emulsions) back into the 2 L separatory funnel, pour the methylene chloride (bottom) layer into a 100 mm filter funnel plugged with glass wool and containing approximately 20 g of sodium sulfate. Collect the filtrate in the 250 mL Erlenmeyer flask. Rinse the funnel with approximately 20 mL methylene chloride and also collect in the 250 mL flask.

13.9 At least 80% of the extraction solvent used must be recovered from a sample that emulsifies, taking into account the solvent's water-solubility. For 100 mL of methylene chloride, at least 70 mL must be recovered (~10 mL is soluble in 1000 mL of water), not counting rinse volumes. For 60 mL, at least 50 mL must be recovered (the water is already saturated from the first extraction), not counting rinse volumes. If at least 80% of the solvent volume is not recovered, narrate on the extraction summary for the instrumental analyst that sample emulsion interference prevented a complete extraction.

13.10 Extract the sample twice more with 60 mL of methylene chloride, rinsing the sample bottle with the first of the two 60 mL aliquots. Repeat Sections 13.8/13.9 as needed. Combine all extracts in the 250 mL Erlenmeyer flask.

13.11 After the extraction is complete, dry the combined extract by pouring through a funnel containing filter paper and about 20 g of sodium sulfate. Collect in a K-D concentrator. Rinse the Erylnmeyer flask with methylene chloride, pour through the funnel and complete the transfer by rinsing the funnel with an additional 20-30 mL of methylene chloride.

Note: A separate extraction blank and blank spike are required if phase separation paper is used for a sample.

13.12 After completing the extract transfer, add a clean boiling chip to the K-D and attach a 3-ball Snyder column to the top. Pre-wet the column with about 1 mL of methylene chloride.

Note: The concentration step is critical as the loss of analytes can occur if care is not taken to prevent the extract from going to dryness.

13.13 Place the K-D in a water bath set at 55-60° C so the concentrator tube is partially immersed in hot water and the entire lower rounded surface is bathed in steam. At the proper rate of distillation, the balls of the column will actively chatter but the chamber will not flood. When the apparent volume reaches no lower than 1 mL, remove the K-D from the bath and allow it to drain and cool for at least 10 minutes.

Note: Do not remove the Synder column until the flask has drained and cooled for 10 minutes.

Note: If an extract becomes highly colored or a heavy precipitate forms during concentration, stop the concentration to determine what final volume can be analyzed.

Note: Do not let the extract go to dryness. If it does, the entire extraction will need repeated.

13.14 After the K-D has cooled, remove and rinse the Snyder column into the K-D flask with 1-2 mL of methylene chloride. Remove the flask from the concentrator tube. Be sure to rinse the ground glass joint, as compounds tend to collect on the ground glass.

13.15 Add another clean boiling chip to the concentrator tube and attach a 2-ball micro-Snyder column.

13.16 Pre-wet the column with ~0.5 mL methylene chloride.

13.17 Place the concentrator tube in the water bath, partially immersed in the water.

13.18 Be sure the water temperature is 55-60° C. Care must be taken to avoid bumping of the extract or flooding of the column chambers. Raise or lower the end of the tube in the water as needed to achieve the proper rate of evaporation. The balls of the column will actively chatter but not flood at the appropriate rate.

13.19 When the apparent volume of extract reaches 0.5 mL, remove the concentrator tube and cool for 10 minutes.

Note: Do not remove the micro-Snyder column before cooling.

Note: Do not let the extract go to dryness. If it does, the entire extraction will need repeated.

13.20 After cooling, remove the micro-Synder column and rinse the lower joint with ~0.2 mL methylene chloride.

13.21 Transfer the extract to a pre-calibrated 1 mL vial using a clean, disposable, Pasteur pipette.

13.22 Rinse the concentrator tube with ~0.2 mL of methylene chloride and transfer to the vial.

13.23 Adjust the final volume in the vial to 1.0 mL with methylene chloride then immediately cap tightly.

13.24 Store all extracts at 4 ±2° C in the GC laboratory refrigerator.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Analysts are responsible for data quality and for correctly filling in all paperwork, laboratory notebooks and other documentation. This is required for quality assurance and to provide the client with fully traceable and defensible data.

14.2 Input all required extraction data to the laboratory information management system (LIMS/Element™).

14.3 Batch reports/extraction summaries must be filled in completely to insure that results are reported correctly and data is associated with the batch quality control.

14.4 If internal chain-of-custody (CoC) is required, it is important that the CoC form be filled in completely and correctly.

14.5 Logbooks must be filled in completely and correctly. **Corrections are to be made with a lineout, initials, and date, not a write-over**. Blank lines in the logbook must be Z'd out.

15.0 QUALITY ASSURANCE

15.1 Extract an extraction blank (BLK), blank spike (BS/LFB), matrix spike (MS) and matrix spike duplicate (MSD) for each extraction batch of up to 20 samples or each day samples are extracted. Whichever is more frequent.

Note: If no sample volume is available for the MS/MSD, extract and evaluate a blank spike duplicate.

15.2 Add 200 µL of 200 ug/mL surrogate to all extractions including extraction blanks (BLK), blank spikes (BS/LFB) and matrix spikes (MS/MSD).

15.3 Prepare the extraction blank by extracting 1000 mL of pre-extracted laboratory reagent water as a sample. Extract and evaluate as a sample.

15.4 Prepare the blank spike by adding 500 µL of 2000 ug/mL DRO spiking solution to 1000 mL of pre-extracted laboratory reagent water. Extract and evaluate as a sample with a 1000 ug/L spike.

$$\frac{2000\ \mu g\ \#2\ diesel\ fuel}{mL} \Big| \frac{0.5\ mL}{1000\ mL} = 1000\ ug/L\ \#2\ diesel$$

15.5 Prepare matrix spikes by adding 500 µL of 2000 ug/mL DRO spiking solution to 1000 mL of sample before extracting. Extract and evaluate as a sample with a 1000 ug/L spike.

15.6 An out-of-control blank spike requires the generation of a non-conformance report to initiate root-cause analysis and corrective action. These need coordinated with the quality assurance department.

15.7 Address all quality control issues in accordance with TriMatrix SOP GR-03-101.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a successful Initial Demonstration of Capability (IDC) study.

16.2 Perform the IDC as follows:

16.2.1 Prepare a 2,000 ug/mL DRO blank spiking standard from a source other than that used for calibration of the DRO gas chromatograph (GC) instrument.

16.2.2 Set up four 1000 mL blank spikes, spike with 500 µL of the spiking standard (and surrogate), and extract as samples.

16.2.3 Analyze in accordance with TriMatrix SOP GR-03-122 then input results to the IDC spreadsheet located on the laboratory intranet library. Average percent recovery must fall within LIMS blank spike acceptance limits. Relative standard deviation must be ≤20%.

16.3 If either criterion is not met, locate and correct the source of the problem and repeat the study successfully.

16.4 Repeated IDC failure will indicate a problem with the procedure and/or techniques used. If this occurs, locate and correct the source of the problem, revise the procedure and/or techniques used and repeat the study successfully.

16.5 Samples may not be extracted by the analyst until an IDC study has been successfully completed.

16.6 Copies of successful IDC studies (spreadsheet and extraction data) must be submitted to the quality assurance department for training documentation.

16.7 A Continuing Demonstration of Capability (CDC) study is required annually. The CDC can be accomplished by any of the following approaches:

16.7.1 By repeating the IDC study.

16.7.2 By using the last four results from the annual method detection limits (MDL) study if extracted exclusively by the analyst.

16.7.3 By inputting four consecutive blank spike results obtained during the course of routine sample processing to the IDC spreadsheet if extracted exclusively by the analyst.

16.7.4 By exclusively and successfully extracting a blind PT study sample during the course of routine sample processing.

16.8 A Method Detection Limit (MDL) study must be extracted annually in accordance with TriMatrix GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update IV, Revision 3, February, 2007, Method 8015C, Nonhalogenated Organics by Gas Chromatography

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision 3, December, 1996, Method 3510C, "Separatory Funnel Liquid-Liquid Extraction".

20.0 ATTACHMENTS

20.1 Extraction Summary Example

20.2 Standards Log Example

20.3 Preparation Batch Report Example

Attachment 20.1
Extraction Summary Example

TriMatrix Laboratories, Inc.

EXTRACTION SUMMARY

CLIENT: Donald Cann PROJECT-SUBMITTAL #: _____ PREPARED BY: DCG

PARAMETER: EPA DRO

EXTRACTION METHOD: 1) Liquid/Liquid 2) Sonication 3) Dilution 4) ASE

EXTRACTION LOCATIONS: BNA: _____ Pest/PCB: _____ Herb: _____ Other: _____

SAMPLE NAME	SAMPLE #	DATE EXTRACTED	WATERS			SOILS		
			mL EXTRACTED	FINAL VOLUME (mL) ~~EPA BNA DRO~~	PEST/PCB	g EXTRACTED	FINAL VOLUME (mL) BNA	PEST/PCB
	CERT 1	6-30-08	1000	1.0				
	CERT 2	↓	↓	↓				
	CERT 3							
	CERT 4							

SPIKING INFORMATION

TYPE	SURROGATE STANDARD ID	AMOUNT (uL)	SPIKE STANDARD ID	AMOUNT (uL)
BNA				
PEST				
PCB				
HERB				
EPA OTHER DRO	806041	200	806040	500

NOTES

09R_Extraction Summary.xls

Approved By: _____ 9-5-08 QA Officer

Approved By: ASC 9/9/08 Area Supervisor

Attachment 20.2
Standards Log Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

7030944

Description:	(AMP) Diesel Fuel #2 2o	Expires:	Apr-23-08
Standard Type:	Other	Prepared:	Aug-31-06
Solvent:	Solvent Lot #A046397	Prepared By:	** Vendor **
Final Volume (mls):	1	Department:	Expired
Vials:	1	Last Edit:	May-13-08 13:05 by JLW

Restek part#31259
Lot: A046397
Received 3/27/07

Analyte	CAS Number	Concentration	Units
DRO - 8015B (C10-C28)		50000	ug/mL

Approved By: _____ 9-5-08 **Approved By:** ASC 9/9/08
QA Officer **Area Supervisor**

Attachment 20.3
Preparation Batch Report Example

TriMatrix Laboratories, Inc. **PREPARATION BATCH** 0809092 Page 1 of 1 Printed: 8/28/2008 2:23:49PM

Semivolatiles GC, Soil, 3550B Sonication Extraction

Batch comments:
(none)

Lab Number	*Container*	*Prepared*	*By*	*Initial (g)*	*Final (mL)*	*Client* Source ID	*Client* Spike ID	*Client* uL Spike	*Comments*	
0807485-19	A	Aug-06-08 08:09	ASC	20.2	1	Environmental Resource Associates			diesel range organics (GRO)	diesel range organics (GRO)
DRO EPA 8015B										
0809092-BLK1		Aug-06-08 08:09	ASC	30	1					
0809092-BS1		Aug-06-08 08:09	ASC	30	1		8060407	500		
0809092-BSD1		Aug-06-08 08:09	ASC	30	1		8060407	500		

Comments:	Analyst Initials:

bch_TM_byAnalysis.rpt

Approved By: QO 9-5-08 **QA Officer**

Approved By: ASC 9/9/08 **Area Supervisor**

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Sample Receipt and Log-in

APPROVALS:

Area Supervisor: _____ Date: 12/15/08
Gary L. Wood

QA Officer: _____ Date: 12-15-08
Tom C. Boocher

Operations Manager: _____ Date: 12/15/08
Jeff P. Glaser

Procedure Number: GR-15-100
Revision Number: 3.2

Date Initiated: 2/27/96
Effective Date: 12/31/08

Date Revised: 12/15/08
Pages Revised: All

By: Gary L. Wood

Total Number of Pages: 32

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SUMMARY OF PROCEDURE

1.1 This document describes in detail the procedures followed during the sample receipt and log-in process at TriMatrix Laboratories. This procedure begins with the arrival of samples at the laboratory and does not end until the samples have been properly stored.

1.2 Samples are received by the laboratory from 8:00 am to 5:00 pm, Monday through Friday. With advance notice, samples can be received on Saturday from 8:00 am to 12 noon. Integrity checks, custody transfer and placement in cold storage will be performed on Saturday. However, a complete sample log-in will not be done until the following business day.

1.3 Sample receipt at other times must be arranged through a project chemist or the log-in staff.

1.4 Technicians receiving samples that require internal chain-of-custody must also review and adhere to TriMatrix SOP GR-10-104.

2.0 DETAILED PROCEDURE

2.1 There are four distinct steps to the sample receipt/log-in process.

Step 1 The initial step acknowledges receipt of samples by the laboratory. The Chain-of-Custody (COC) is signed and required entries are made in the Sample Receipt Records Logbook. Step 1 must be completed immediately, at the time of sample receipt. However, the cooler is not opened during Step 1.

Step 2 A Sample Receiving/Log-In Checklist is initiated. The cooler is inspected for intact custody seals then opened. The sample type, sample quantity and coolant location are documented, and the sample temperature is taken and recorded. When necessary, a Sample Receiving Non-Conformance Report is initiated.

IMPORTANT: Never open a cooler unless planning on immediately completing Step 2 of the receipt/log-in process.

Step 3 Samples are removed from the cooler during the third step of the receiving process. Upon removal, samples are physically inspected and compared to the COC. The preservation of chemically preserved samples is verified and the laboratory is informed of any short hold time analysis requirements. All initial paperwork is collected and delivered to the project chemist.

Step 4 The final step of the sample receipt/log-in process involves performing any authorized pH adjustments, logging samples into the LIMS system and storing samples in assigned locations.

2.1.1 To safely and correctly complete the sample receipt/log-in process, the following equipment is required:

- Laboratory coat
- Approved safety glasses
- Disposable gloves
- Infrared thermometer, Raytek Raynger, model ST60XX95, verified as NIST-traceable

Alternative: stem-type digital thermometer, verified as NIST-traceable
Alternative: liquid-in-glass thermometer, verified as NIST-traceable
- ColorpHast strips, pH 0-14 universal, EMD catalog 9590 or equivalent
- Preservation reagents, contaminant-free
- Fume Hood

2.1.2 Personal protective equipment must be worn during sample receipt/log-in. A laboratory coat and approved safety glasses must be worn in the sample receipt and bottle preparation area except when entering data at a computer terminal.

2.1.3 Many received coolers are quite heavy. A back brace is available and highly recommended when lifting and moving any heavy object. A wheeled table must be used when transporting coolers from the garage into the sample receipt/log-in area.

2.1.4 For samples known to be extremely hazardous or toxic, additional safety precautions are required and noted throughout the procedure. Notify the Health and Safety Officer and Project Chemist before sample receipt/log-in of non-routine hazardous or toxic samples unless otherwise specified.

2.1.5 No food or drink is permitted in the sample receipt/log-in area at any time, including chewing gum.

2.2 Step 1 is outlined as follows:

2.2.1 Immediately upon receipt of a sample or sample cooler, the receipt must be recorded in the Sample Receipt Record logbook (Attachment 6.1). The logbook must contain the following entries:

- Delivery method
- Date received
- Time received
- Number of coolers received
- Client
- Name of the technician receiving coolers and recording the information

2.2.2 The "received by", date and time must also be used to complete the "received for lab by" section of the field Chain-Of-Custody (COC) form (Attachment 6.2).

2.2.3 Samples arriving via walk-in clients or TriMatrix field services personnel must be accompanied by a COC form. When possible, have the delivery person sign in the first available "relinquished by" section on the COC. The TriMatrix technician receiving the samples must sign the "received for lab by" section. This information must match that entered in the Sample Receipt Logbook. Return the pink COC copy to the delivery person.

NOTE: It is possible that samples delivered by a TriMatrix employee will be "relinquished by" and "received for lab by" the same person. This can occur after normal receiving hours or on weekends when log-in staff are not available.

NOTE: The number of coolers delivered by different delivery methods but from the same project-submittal must be additively entered into a column of the Sample Receipt Record logbook as the total number of coolers received for the project. This number must be verified when the logbook is checked, by counting to confirm the number of coolers received from all delivery methods is the same as the number recorded.

2.2.4 If Step 2 will not immediately begin, the sample cooler must be placed in the walk-in cooler.

NOTE: When samples requiring temperature preservation are received at ambient temperature, Step 2 must be performed immediately. Refrigerating non-chilled samples prior to recording their temperature results is a misrepresentation of the preservation status of the sample.

2.3 Step 2 is outlined as follows:

2.3.1 Observations associated with any step of the process must be recorded on the Sample Receiving/Log-In Checklist (Attachment 6.3). The Checklist must be completed for all samples in a given project received during the day. A Sample Receiving/Log-In Checklist Additional Cooler Information is available when receiving samples in a given project consisting of more than four coolers (Attachment 6.4).

NOTE: Shaded boxes on the Checklist indicate an out-of-control situation. The selection of ANY shaded box during Checklist completion requires initiation of a Sample Receiving Non-Conformance Report (Attachment 6.5).

2.3.2 When sample coolers are received, segregate by project. Complete the upper two Checklist sections to the extent possible. To maintain cooler temperature integrity, open and determine sample temperature on one cooler at a time.

NOTE: It is important when initiating a Checklist to be sure the page/line number from the Sample Receipt Record logbook is recorded at the top of the Checklist. The number associates a sample cooler with the samples received in the cooler.

2.3.3 Document whether custody seals are present and their condition. Remove and retain all custody seals, then open the cooler.

NOTE: Opening a cooler known or suspected to contain highly hazardous or toxic samples MUST be performed in the fume hood.

2.3.4 Before discarding any packing material collect all paperwork from inside the cooler. Paperwork sent with samples can contain significant project information, including but not limited to field screening readings, general field information, and hazard warnings. These must be retained.

2.3.5 Record the cooler number and the time the cooler was opened.

2.3.6 Observe and record the type and location of coolant used.

2.3.7 Temperature Measurement

2.3.7.1 When possible, measure and record the temperature of three representative samples from random locations within the cooler. Also, measure and record the temperature blank, if one is included.

2.3.7.2 Sample temperatures are measured using a calibrated infrared (IR) thermometer (Section 4.6). The IR thermometer measures sample temperature indirectly by taking a surface reading. Consequently, IR readings MUST be taken just as a sample is lifted from the cooler. Sample container surfaces warm quickly and any other technique will result in an incorrect measurement. Do not dry the container before taking a reading. Containers wet from melt-water are more representative when wet than those not having been in direct contact with coolant. However, do not replace a sample chosen at random that does not have melt-water. It must be used.

2.3.7.3 Aim the IR thermometer approximately six inches from the surface to be measured and pull the trigger. On clear glass containers aim the laser through the container onto the back of the label. This is not required with opaque containers (measurements can be taken without a label shot).

2.3.7.4 Laser aiming from the thermometer will illuminate the surface being measured. Hold the trigger in for 3 to 5 seconds then release and observe the result shown on the screen. Record the result obtained in degrees centigrade.

2.3.7.5 If the infrared thermometer is not available, a digital or liquid-in-glass thermometer may be used, if verified as NIST-traceable (a tag will be attached indicating when verification was last performed). These thermometer types have a potential for contamination if used directly on samples. Consequently, temperature readings must be taken only on a temperature blank or the coolant melt-water. To take a digital or liquid-in-glass thermometer reading, immerse the stem in an opened temperature blank or directly in coolant melt-water. Observe and record the temperature after the reading stabilizes. Do NOT open samples to take a temperature reading. See the quality assurance manager for assigning an alternative thermometer to the department when needed.

2.3.7.6 Record individual temperature readings and the average from each cooler on the Sample Receiving/Log-In Checklist. Report all values to the nearest 0.1° C. If a correction factor is necessary (based on the daily NIST traceability verification), record the correction factor and the corrected temperature on the Sample Receiving/Log-In Checklist.

2.3.7.7 If a temperature average or temperature blank exceeds 6° C, note on the Sample Receiving/Log-In Checklist. A Sample Receiving Non-Conformance Report will be required.

2.3.7.8 The entire procedure must be followed for each sample cooler received.

NOTE: If a trip blank is received, record the cooler number on the trip blank tag.

2.3.8 If sample receipt and temperature determination are performed outside of normal business hours, replace the temperature blank or sample removed for the temperature determination in the original cooler and transfer to the laboratory walk-in cooler. Collate all paperwork and place in the after-hours basket. The receiving process will continue on the next business day. Otherwise, proceed to Step 3.

NOTE: Accompanying paperwork including bills of lading, packing slips, air bills, manifests, field sampling forms, and correspondence documents must be retained and saved with the COC.

NOTE: If placing the sample cooler in the walk-in, first remove any large quantities of ice present on top of samples before placing in the walk-in. This will prevent damage to sample identification tags from residual melt-water. The walk-in will keep samples at an acceptable temperature.

2.4 Step 3 is outlined as follows:

2.4.1 Verify that the COC "received for lab by" is filled in. If not, it must be done immediately. To maintain the chain-of-custody timeline, enter the initials, date and time recorded in the Sample Receipt Record logbook. If verification is done by the person who received samples, record a signature (not initials) on the COC. Complete the "paperwork received" and "COC ID Nos." section of the Sample Receiving/Log-In Checklist.

2.4.2 The COC must be reviewed for accuracy. Complete the "check COC for accuracy" section of the Sample Receiving/Log-In Checklist. Additional information that must be present and correct on the COC include:

- Client name, address and contact phone number, unless on file
- Number of containers per sample
- Requested analyses (if not initially present, will be filled in by project chemist via the client)
- Page/Line number from the Sample Receipt Record logbook

2.4.3 Sample collection personnel should have filled in sample label information. If the information is missing or incomplete, notify the project chemist. In many cases, missing sample label information can be determined after checking label information on remaining samples against the COC. However, assignment of client-based sample information by TriMatrix receiving technicians must first be brought to the project chemist's attention and documented on a Sample Receiving Non-Conformance Report.

NOTE: Additional information may also be supplied by the COC, depending upon project specifications and COC type (TriMatrix or client). The project chemist must evaluate what additional information is pertinent to the log-in process and communicate this to applicable log-in staff.

2.4.4 Affix appropriate labels onto sample containers. These include MS/MSD, USDA-regulated, Composite Before Analysis, PT Sample, Caution!!!, and Return Sample to Waste Cabinet labels (Attachment 6.6).

2.4.5 Complete the "sample condition summary" section. If sample containers were broken during shipment and/or uncontained sample is observed in a cooler, unless it is known that the samples contained in the cooler are not hazardous, implementation of the following steps is required.

2.4.5.1 Immediately reseal the shipping cooler and place in the sample receipt room hood. If a cooler is too large for the hood, place in a well-ventilated area in the garage. Place a note on the cooler that it may contain contamination and is not to be opened.

2.4.5.2 Immediately notify the project chemist and when appropriate the health and safety officer. Do not continue with sample log-in for the affected samples until sufficient safety information is obtained.

2.4.5.3 The project chemist must consult with the client on how to proceed and when appropriate, request safety information. If instructed to proceed with the log-in process, review the safety information obtained and observe appropriate safety precautions. Remove the cooler contents, properly dispose of any adsorbent and decontaminate the inside and outside. Record all resolutions on the Sample Receiving Non-Conformance Report.

2.4.6 In addition to COC and cooler/sample discrepancies, project-specific issues may also be observed. Any aberration from acceptable sample receipt protocol must be documented on a Sample Receiving Non-Conformance Report when found. Such issues include the following:

- Project name correctness
- Hold time violations due to late receipt
- Illegible paperwork
- Incorrect project or purchase order numbers
- Missing project chemist name
- Questionable parameter list
- General information missing

2.4.7 Many aqueous samples received are subjected to some form of chemical preservation. Verification of the preservation is required. Depending on the analysis, preservative verification may not occur during log-in. The Sample Preservation Verification Form (Attachment 6.7) specifies which sample types will have preservation verified during log-in. The form also specifies which preservation may be adjusted by log-in, if samples are received incorrectly. No preservation adjustment may be made without project chemist approval.

2.4.7.1 Acid and base preservation is checked using 0-14 pH strips. Determine sample pH by briefly dipping (1-3 seconds) a pH strip in a sample and comparing the pH strip color against the chart on the pH strip container. The color that matches is the pH reading. Record preservation verification results on the Sample Preservation Verification Form. If preservation is acceptable, place a check mark in the box corresponding to the checked sample. If preservation is not acceptable, record the actual pH obtained in the appropriate box. Use only pH strips located in the log-in room. Each lot is verified for accuracy and tested as contaminant-free (section 4.3).

IMPORTANT: It has been previously documented that over time barium will leach off a pH strip. Because of the potential for barium contamination, when testing the pH of a number 6 container, never allow the pH strip to remain in the water longer than 3 seconds.

NOTE: If there is insufficient evidence that other preservation (for example, zinc acetate for sulfides) is present, a Sample Receiving Non-Conformance Report must be initiated and the project chemist contacted immediately.

2.4.7.2 If a preservation check indicates unacceptable pH at sample-receipt, an orange Out-of-Control Sample pH tag (Attachment 6.8) must be placed on the container and "initial pH" filled in. In some cases, the sample hold time will be shortened. Except for type 6 bottles, the pH adjustment will be made in Step 5.

2.4.7.2.1 Acid preservation on type 6 bottles at the time of (or following) sample receipt requires sample pre-treatment or analysis be delayed for a minimum of 16 hours. The delay allows the added acid to re-solubilize precipitated or wall-adsorbed analyte (Refer to TriMatrix SOP GR-01-145, Section 8.1). To avoid additional delay it is important that the metals preparation staff be informed immediately of the preservation adjustment. Provide a copy of the COC and the Sample Preservation Verification Form. Upon receipt of the COC/SPV forms, the metals preparation staff must obtain project chemist approval to perform the pH adjustment.

2.4.7.2.2 The metals preparation analyst must record all adjustment information on the out-of-control pH tag when making an adjustment.

2.4.8 Sample hold time is defined as the maximum storage time allowed between sample collection and analysis when designated preservation and storage techniques are employed. Hold times are typically specified to include any sample preparation as part of the analysis. Sample preparation and analysis must occur within the EPA-prescribed hold time or sample data must be qualified. Consequently, it is important that appropriate laboratory areas be informed immediately of any received samples with a significant time delay between collection and receipt by the laboratory. Also, it is important that appropriate laboratory areas be informed immediately of any received samples with very short hold times.

NOTE: It is the log-in technician's responsibility to be familiar with hold times and to identify any received sample parameter with a short hold time. A Short Hold Time Analysis List is provided in Attachment 6.9.

2.4.9 If samples with short hold times are received, check the appropriate box or boxes on the Sample Receiving Log-In Checklist and notify the project chemist and all applicable laboratory chemists. Document the completion of all notifications on the Checklist. Provide each affected laboratory area with a copy of the COC.

Approved By: 12-15-08 **QA Officer** **Approved By:** 12/15/08 **Area Supervisor**

2.4.10 If a sample is received with an expired hold time or with a hold time due to expire during sample log-in, contact the project chemist immediately.

2.4.11 Samples must be placed in temporary storage until the Laboratory Information Management System (LIMS) data entry is complete.

2.4.11.1 Water and wastewater VOC vials, charcoal tubes and airbags must be stored in the water-volatiles refrigerator located in the log-in area. Samples requiring refrigeration must not be left un-refrigerated longer than 15 minutes after unpacking. Bulk soil VOC containers must be stored in the soil-volatiles refrigerator, located in the log-in area. EnCore®, methanol-preserved and bisulfate-preserved volatiles soil samples must also be stored in the soils refrigerator. However, due to short hold times these are typically delivered and stored in the appropriate refrigerator located in the volatiles laboratory. Received microbiological samples must be stored on the designated shelf in the walk-in cooler.

2.4.11.2 All waste samples must be placed in the log-in hood when received. Waste-volatile samples must be stored inside the waste-volatiles refrigerator located in the log-in hood.

NOTE: If a received waste sample is to be analyzed for volatiles but no dedicated sample container is received, inform the laboratory. Have an analyst remove an aliquot for the volatiles analysis before the sample is opened for other analyses.

2.4.11.3 All other water, wastewater and soil containers must be placed on a numbered cart and stored in the walk-in refrigerator. Record the cart number on the Chain-of-Custody to help laboratory chemists locate short hold time samples that have not been logged in.

2.4.12 Additional information or questions for the project chemist can be recorded in the "notes" section of the Checklist. Record whether a trip blank was received/not received. If received, mark that the trip blank was recorded on the COC. Record the date and time the project was received at the laboratory and the time paperwork was brought to the project chemist.

2.4.13 All paperwork received with samples such as bills of lading, packing slips, air bills, manifests, field sampling forms, correspondence documents, the COC and all other paperwork generated during the log-in process must be placed in a green folder and delivered to the appropriate project chemist for an initial paperwork review. If the project chemist is not in the office, place the folder in the project chemist's bin.

2.4.14 The project chemist must review all paperwork, create a Sample Login Specification Form and return all paperwork to the log-in area.

2.5 Step 4 is outlined as follows:

2.5.1 After reviewing the paperwork and creating the Sample Login Specification Form, the project chemist must return the green folder to the log-in area. The assigned technician must then

review page two of the Sample Receiving/Log-In Checklist for any additional instructions made by the project chemist. Using the Sample Receiving/Log-In Checklist, the COC and any associated receipt paperwork the assigned technician may proceed to log samples into LIMS (Element™). Each step of this process must be documented on page two of the Checklist.

2.5.2 The information listed below is required to properly log samples into LIMS. After the information has been entered and compiled, LIMS will print labels for each sample container. Printed labels are affixed to each associated sample container tag.

2.5.2.1 Client and project names - All narratives entered into LIMS by the project chemist must be reviewed.

2.5.2.2 Schedules to be used – All relevant analysis, matrix, container type and size, and quantities must be scheduled into the project by the Project Chemist prior to sample login unless otherwise specified. The login technician may then select the schedule referenced on the COC and detailed on the Sample Login Specification Form. After selecting the schedule, modify the field sample name (if different than the schedule name) then add the sampler's name and the collection date and time from the COC. Each number must be unique and designated as the laboratory identifier for the given sample.

2.5.2.3 Bottle description (as received) - The number corresponding to the bottle-type on the Container Packing List (Attachment 6.10). The Container Packing List is generated from the bottle order that supplied sample collection containers.

2.5.2.4 Client (field) sample identification from the COC.

2.5.2.5 Sample collection date and the military time.

2.5.2.6 When supplied, first initial and last name of the sampler.

2.5.2.7 COC number if available (TriMatrix COC forms are uniquely numbered).

2.5.2.8 The number of containers received for each sample.

2.5.2.9 Vial rack or tray number (for a volatiles sample) and storage area identification.

2.5.2.10 When complete, record the initials/date/time on the Receiving/Log-In Checklist.

NOTE: All trip blanks must be assigned a "date sampled" and "time sampled". Assign a "date sampled" equal to the latest sample date of all the samples in the submittal. Assign a time collected of 00:00.

NOTE: It is common for a client to assign a different collection date and/or time for trip blanks such as the date a trip blank is shipped or no collection time. Any correction to a client-specified collection date and/or time must be made or authorized by the project chemist with the change properly noted on the COC. Refer to Section 4.13 regarding COC corrections.

2.5.3 Perform the following after entry of the above information into LIMS.

2.5.3.1 Affix each printed label to the associated sample tag or container.

NOTE: If there are samples unaccounted for at this time (i.e. a sample is encountered that was not found on the COC, initiate a non-conformance and inform the project chemist. Check the Discrepancy box on the non-conformance form.

2.5.3.2 If approval is given, any necessary preservation adjustments may now be made to orange-tagged samples.

2.5.3.2.1 Only for those container types listed on the Sample Preservation Verification Form and only with project chemist/client approval, add acid or base to samples requiring a pH adjustment. All post-receipt preservations must use reagents from the bottle preparation room. To make a pH adjustment, add just enough acid or base to correct the discrepancy but do not exceed twice the original preservative volume used for normal bottle preparation. Normal volumes are listed on the Sample Preservation Verification Form. If pH is not adjusted using twice the normal volume, the sample exhibits buffering and must be noted as such on the Sample Preservation Verification Form.

2.5.3.2.2 After pH adjustment, record the final pH in the box adjacent to the original pH on the Sample Preservation Verification Form. Also, record the final pH and acid/base volume added in the appropriate box on the orange "out-of-control sample pH" tag. Check "ok" or "still out" relative to the final pH attained.

2.5.3.2.3 The technician performing a pH adjustment must complete the "adjusted by" box on the Sample Preservation Verification Form.

2.5.3.3 Initial, date and check the "label sample containers" section of the Sample Receiving/Log-In Checklist after labeling.

2.5.3.4 All tagged and labeled sample information must be verified by initialing and dating the "verified by" section of the Checklist.

2.5.4 Sample Storage

2.5.4.1 All samples must be stored in a secure restricted-access area and/or unit.

2.5.4.2 Samples are stored in the walk-in cooler shelf areas according to sample number, in a designated refrigerator or in numbered container racks.

2.5.4.3 Sample storage refrigerator information is recorded in the Controlled Temperature Unit (CTU) Daily Monitoring Logbook.

2.5.4.4 No sample may be stored in the same refrigerator as analytical standards, chemicals or food/drink. These items are prohibited from storage in any refrigerator designated for sample storage.

2.5.4.5 Volatiles samples are stored in a refrigerator in the volatiles laboratory. Soil and water samples are stored separately. Samples are further organized by shelf and rack number.

2.5.4.6 Non-volatile water and soil samples are stored in the walk-in cooler. Waste samples for volatiles analysis are stored in a designated refrigerator in the sample receipt/log-in fume hood.

2.5.4.7 Non-volatile waste samples are stored in a vented storage cabinet in the garage area. Samples are segregated by hazard class. The client name and sample number of each sample stored must be written on the Waste Cabinet Sample Inventory Form (Refer to Attachment 6.11).

2.5.4.8 If a received sample is determined to be extremely hazardous or toxic during log-in or analysis, all aliquots of the sample must be relocated to the waste storage cabinet.

2.5.4.9 Tedlar® bags and charcoal tubes for air analysis are stored in the log-in water-volatiles refrigerator.

2.5.4.10 Microbiological samples are stored on a designated shelf in the walk-in cooler.

2.5.4.11 As sample numbers change in the walk-in refrigerator, storage shelf labels must be clearly marked with the new range of numbers.

2.5.5 Sub-sample compositing is performed by applicable laboratory analysts when needed. A composite sample must be identified as such on the composite container tag. A LIMS narrative must also be entered by the compositing analyst.

2.6 LIMS-assigned Laboratory Sample Identifier (ID)

2.6.1 A unique LIMS-assigned laboratory sample identification number (ID) is generated during the log-in process. The sample ID consists of the workorder number followed by a hyphen and a numeric identifier unique to each sample received (for example: -01, -02, -03).

2.6.2 The workorder number consists of the truncated year and month followed by a three-digit number representing how many workorders were received month-to-date. For example, 0704456 reflects the year 2007, April and the 456th workorder logged in during April, 2007.

2.6.3 The workorder number appears on laboratory benchsheets and is used as the sample ID on all instrument run logs and associated raw data. It is traceable to the client-assigned sample identifier.

2.6.4 A letter suffix follows any sample ID to uniquely identify each sample container received. For example, 0704456-04A identifies the first sample container received; 0704456-01B identifies the second and so on until all sample containers are identified.

2.7 At the completion of the log-in process, collect all chain-of-custody forms and other documents received, the Sample Receiving/Log-In Checklist, all other log-in documents and the LIMS-generated workorder report (once available) in a folder. Deliver the folder to the project chemist for review and approval.

2.8 Sample Disposal is as follows:

2.8.1 The number of days samples must be retained by the laboratory after the completion of all analyses is specified in LIMS by the project chemist. The LIMS default is 14 days after the final data report has been generated.

2.8.2 When the sample retention time has expired, samples may be appropriately disposed of (non-hazardous or hazardous) or returned to the client.

2.8.3 The waste disposal technician or other designated person must determine how samples are disposed of in accordance with the latest revision of TriMatrix SOP GR-15-102.

3.0 REPORTING AND DELIVERABLES

3.1 There is no reporting and deliverables directly associated with this procedure other than those described above.

4.0 QUALITY ASSURANCE

4.1 For each workorder generated, workorder numbers must be entered into the Sample Receipt Record Logbook. Verification that a workorder folder has been compiled must also be documented by recording in the "folder prepared" column of the logbook. This logbook must be reviewed on a daily basis.

4.2 Each lot of pH strips must be checked against certified pH buffers and documented in the pH Strip Calibration Logbook (Attachment 6.12). This test is performed by the wet chemistry laboratory.

4.3 New pH strip lots must also be tested for contaminants and interferences.

4.3.1 When an approximately one-month supply of pH strips remains, place an order for another four to six-month supply. The ordered pH strips must be from one lot number. Upon receipt by the laboratory, provide the Quality Assurance Department with the lot number. The Quality Assurance Department will act as the project chemist and create a LIMS workorder for the test.

4.3.2 To test a new pH strip lot, add the appropriate preservative to container types 3, 4, 5, 6, 13 in accordance with TriMatrix SOP GR-15-101. Fill with laboratory reagent water from the applicable laboratory area and deposit a pH strip in each container. Let the containers stand for 24 hours in the sample receipt/log-in fume hood.

NOTE: It has been previously documented that barium will leach from a pH strip during this test. For that reason, prepare a second type 6 bottle without the addition of a pH strip. Rather than depositing the strip in the container the metals analyst must quickly dip the strip in the container to mimic a typical pH sample check. Analyze the water in the dipped container for barium only.

4.3.3 After 24 hours, deliver all test containers to the appropriate laboratory area for analysis. The following analyses must be performed on the test water:

Container Number	Tag Color	Preservative	Test
5	Light Blue	NaOH	Cyanide
3	Green	none	Nitrate, Nitrite, Sulfate, Chloride, Fluoride, Bromide, Cr^{6+}, Alkalinity, Acidity, o-Phosphate
4	Dark Blue	H_2SO_4	TKN, Nitrate-Nitrite, Phosphorus, COD, Ammonia
13	Brown	H_2SO_4	Phenolics
6	Red	HNO_3	Ca, Fe, Li, Mg, Hg, K, Na, Sr, Sn, Ti, Al, Sb, As, B, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Tl, V, Zn
6	Red	HNO_3	Ba

4.3.4 The Quality Assurance Officer must review the test results and approve/reject the new lot of pH strips. Do not use the new lot until approval has been received.

4.4 Liquid-in-glass thermometers must be verified against a NIST-traceable thermometer, at least annually.

4.5 Digital stem-type thermometers must be verified against a NIST-certified thermometer, at least quarterly.

4.6 The Infrared thermometer must be verified daily against the NIST-traceable thermometer located in the walk-in cooler. This verification is performed by the Quality Assurance Department. The IR reading must agree within ±0.5° C of the cooler thermometer. Document the test results in the Infra-Red Thermometer Calibration Record logbook (Attachment 6.13). A difference of greater than ±0.5° C will require the use of a correction factor on all temperature measurements made that day.

4.7 Refrigerator temperatures must be verified daily. Results of the verification are recorded in the Controlled Temperature Unit Logbook (Attachment 6.14). This verification is performed by the Quality Assurance Department.

4.8 Storage blanks are in all refrigerators used for volatile organic samples storage. These blanks are analyzed weekly by the volatiles laboratory.

4.9 All tagged and labeled sample information must be verified by initialing and dating the "verified by" section of the Checklist

4.10 Paperwork contained in the project submittal receipt document folder must be reviewed by the project chemist.

4.11 Samples whose pH preservation is incorrect must be tagged with an out-of-control sample pH tag. Improper preservation is corrected only with approval from the project chemist. Record the discrepancy on the tag and all appropriate paperwork (Section 2.4.7).

4.12 A Sample Receiving Non-Conformance Report must be generated for the following problems:

- A sample ID discrepancy between a COC and container
- Missing, broken, inappropriate or extra container
- Illegible, missing or incomplete label
- Headspace in a container
- Low sample volume
- Incorrect preservation (chemical and/or temperature)

4.13 Irregularities and non-conformances in sample receipt/log-in documentation must be appropriately corrected and fully documented. The chain-of-custody and associated documents comprise an agreement between TriMatrix Laboratories and clients for analytical services requested and performed.

4.13.1 If a COC item requires correction, draw one line through the item then date and initial the line. This must be done only by client request or authorization. Consequently, always include "per <Client representative's name>".

4.13.2 If a client provides pre-digested, pre-leached or pre-extracted samples and is requesting only an analysis, notify the project chemist before proceeding with sample log-in. The exception is when prior arrangements were made and previously communicated to log-in by the project chemist.

4.13.3 Do NOT obliterate an original communication by scribbling or any other type of write-over. The original must remain legible. Strike out the original communication with a single line then date and initialize the correction.

4.13.4 Use ONLY indelible blue or black ink.

5.0 REFERENCES

5.1 TriMatrix SOP GR-15-101, *Bottle Preparation*, latest revision

5.2 TriMatrix SOP GR-10-104, *Internal Chain-of-Custody*, latest revision

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.4 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision 3, December, 1996, Chapter 2 Table 2-36, Chapter 4 Table 4-1, Chapter 3 Table 3-1

Approved By: _____ 12-15-08 **QA Officer** **Approved By:** _____ 12/15/08 **Area Supervisor**

5.5 *Code of Federal Regulations*, Title 40, Protection of the Environment, Volume 19, latest edition, Chapter I, Part 136, Subpart D, Guidelines Establishing Test Procedures for the Analysis of Pollutants, Environmental Protection Agency

5.6 *Code of Federal Regulations*, Title 40, Protection of the Environment, Volume 19, latest edition, Chapter I, Part 141, Subpart D, National Primary Drinking Water Regulations

5.7 Competency Requirements for Calibration and Testing Laboratories: International Standards Organization, ISO/IEC Guide 17025, latest revision

5.8 National Environmental Laboratory Accreditation Conference (NELAC), *Quality Systems*, Chapter 5, latest revision

5.9 TriMatrix SOP GR-01-145, *Inductively Coupled Plasma Atomic Emission Spectroscopy*, latest revision

6.0 ATTACHMENTS

6.1 Sample Receipt Record logbook

6.2 Field Chain-of-Custody form

6.3 Sample Receiving/Log-In Checklist

6.4 Sample Receiving/Log-In Checklist-Additional Cooler Information

6.5 Sample Receiving Non-Conformance Report

6.6 TriMatrix Sample Container Tag and Log-In Labels

6.7 Sample Preservation Verification Form

6.8 Out-of-Control Sample pH Tag

6.9 Short Hold Time Analysis List

6.10 Container Packing List

6.11 Waste Cabinet Sample Inventory form

6.12 pH Strip Calibration Logbook

6.13 Infra-Red Thermometer Calibration Record

6.14 Controlled-Temperature Unit Daily Log Sheet

Attachment 6.1
Sample Receipt Record

Sample Receipt Record

TriMatrix Laboratories, Inc.

Date: _____

Delivery Method A: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Delivery Method B: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Delivery Method C: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Delivery Method D: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

TriMatrix Courier (TC): _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

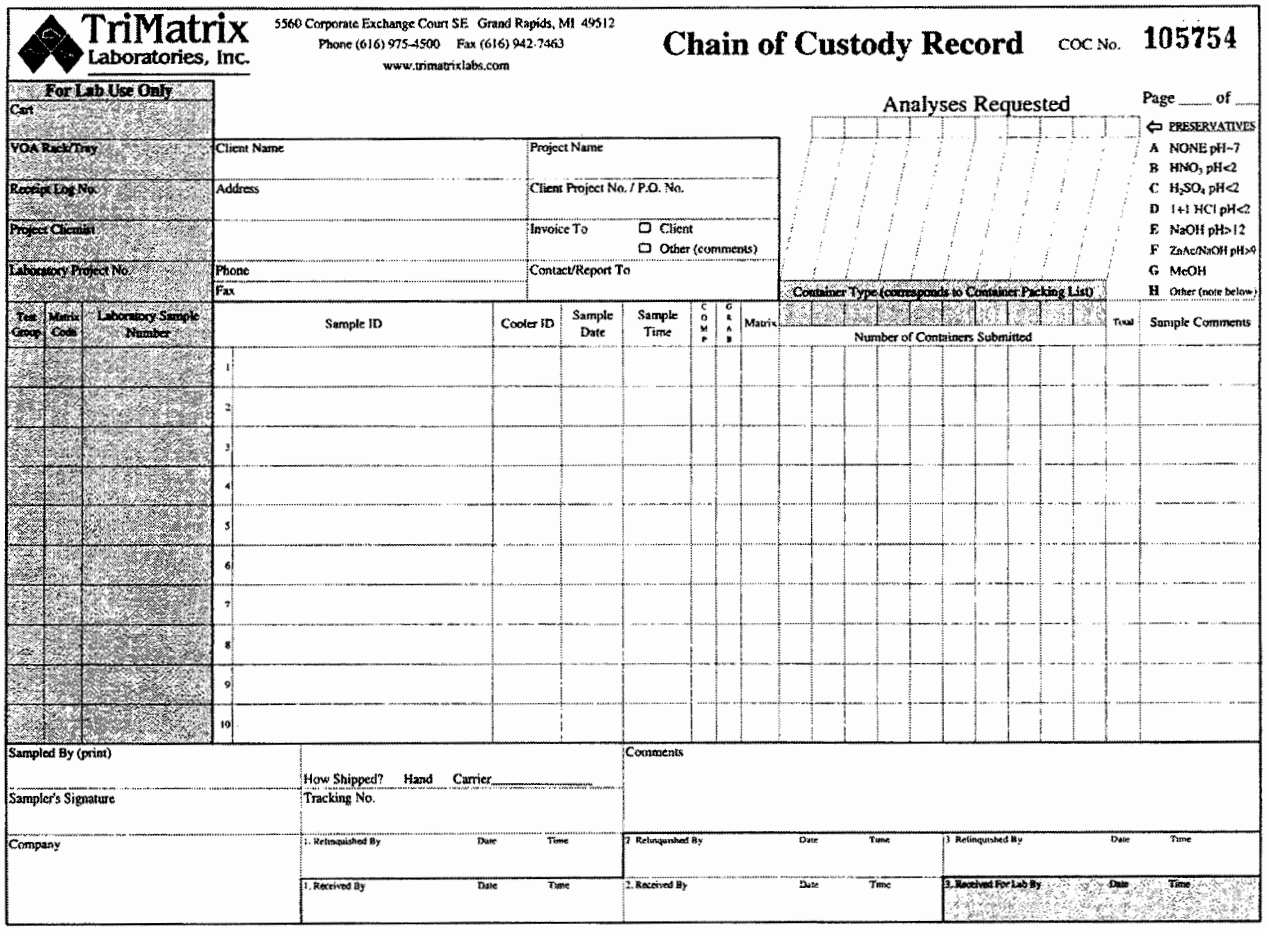
Page/ Line Number	Client	Quantity of Coolers OR TriMatrix Cooler Number	Arrived in Laboratory					Submittal Number (Project Chemist)	Folder Prepared (Log-In ✓)
			Time	AM	PM	Received By	Delivery Method Letter		
1-1									
1-2									
1-3									
1-4									
1-5									
1-6									
1-7									
1-8									
1-9									
1-10									
1-11									
1-12									
1-13									
1-14									
1-15									
1-16									
1-17									
1-18									
1-19									
1-20									
1-21									

file: samplereceipt 2 page: 1 of 10 revision: 1.8

Approved By: _____ 12-15-08
QA Officer

Approved By: _____ 12/15/08
Area Supervisor

Attachment 6.2
Field Chain-of-Custody Record



TriMatrix Laboratories, Inc.
5560 Corporate Exchange Court SE Grand Rapids, MI 49512
Phone (616) 975-4500 Fax (616) 942-7463
www.trimatrixlabs.com

Chain of Custody Record COC No. **105754**

Page ____ of ____

For Lab Use Only
Cart
VOA Rack/Tray
Receipt Log No.
Project Clientint
Laboratory Project No.

Client Name
Address
Phone
Fax

Project Name
Client Project No. / P.O. No.
Invoice To ☐ Client ☐ Other (comments)
Contact/Report To

Analyses Requested

⇦ PRESERVATIVES
A NONE pH~7
B HNO_3 pH<2
C H_2SO_4 pH<2
D 1+1 HCl pH<2
E NaOH pH>12
F ZnAc/NaOH pH>9
G MeOH
H Other (note below)

Test Group	Matrix Code	Laboratory Sample Number	Sample ID	Cooler ID	Sample Date	Sample Time	COMP	GRAB	Matrix	Container Type (corresponds to Container Packing List)	Number of Containers Submitted	Total	Sample Comments
			1										
			2										
			3										
			4										
			5										
			6										
			7										
			8										
			9										
			10										

Sampled By (print)
Sampler's Signature
Company

How Shipped? Hand Carrier_____
Tracking No.

Comments

1. Relinquished By	Date	Time	2 Relinquished By	Date	Time	3 Relinquished By	Date	Time
1. Received By	Date	Time	2. Received By	Date	Time	3. Received For Lab By	Date	Time

TriMatrixCOC -- COC WHITE COPY - REPORT YELLOW COPY - LABORATORY PINK COPY - FIELD

Approved By: _____ 12-15-08
QA Officer

Approved By: _____ 12/15/08
Area Supervisor

Attachment 6.3
Sample Receiving Log-In Checklist (page 1)

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST

Client | Project-Submittal No. (new / add to)
Receipt Record Page/Line No. | Project Chemist | Sample Nos.

Coolers Received

Recorded by (initials/date) | ☐ Cooler ☐ Box ☐ Other | Qty Received | ☐ IR Gun (#94) | Thermometer Used ☐ Digital Thermometer (#54) ☐ Other (#_____) | ☐ See Additional Cooler Information Form

Cooler No. / Time	Cooler No. / Time	Cooler No. / Time	Cooler No. / Time
Custody Seals: ☐ none ☐ present / intact ☐ present / not intact	Custody Seals: ☐ none ☐ present / intact ☐ present / not intact	Custody Seals: ☐ none ☐ present / intact ☐ present / not intact	Custody Seals: ☐ none ☐ present / intact ☐ present / not intact
Coolant Location: Dispersed / Top / Middle / Bottom	Coolant Location: Dispersed / Top / Middle / Bottom	Coolant Location: Dispersed / Top / Middle / Bottom	Coolant Location: Dispersed / Top / Middle / Bottom
Coolant/Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ■ none / avg 2-3 containers	Coolant / Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ■ none / avg 2-3 containers	Coolant / Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ■ none / avg 2-3 containers	Coolant / Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ■ none / avg 2-3 containers
Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container	Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container	Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container	Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container
Recorded °C / Correction Factor °C / Actual °C	Recorded °C / Correction Factor °C / Actual °C	Recorded °C / Correction Factor °C / Actual °C	Recorded °C / Correction Factor °C / Actual °C
tb	tb	tb	tb
tb location: representative / in ice	tb location: representative / in ice	tb location: representative / in ice	tb location: representative / in ice
1	1	1	1
2	2	2	2
3	3	3	3
Average °C	Average °C	Average °C	Average °C
☐ Cooler ID on COC?	☐ Cooler ID on COC?	☐ Cooler ID on COC?	☐ Cooler ID on COC?
☐ VOC trip blank received?	☐ VOC trip blank received?	☐ VOC trip blank received?	☐ VOC trip blank received?

If any shaded areas checked, complete Sample Receiving Non-Conformance Form

Paperwork Received ☐ No COC received

N/A	Yes	No	
	☐	☐	Chain of Custody Record(s)?
	☐		If No, COC initiated by _____
	☐		Rec'd for Lab signed/date/time?
☐	☐		Shipping Document?
☐	☐		Other _____

COC ID Nos.

☐ TriMatrix

☐ Other (name or ID#)_____

Check COC for Accuracy ☐ No analysis requested

Yes	No	
☐	■	Sample ID matches COC?
☐	■	Sample date and time matches COC?
☐		Container type completed on COC?
☐	■	All container types indicated are received?

Sample Condition Summary ☐ Non-TriMatrix containers, see Notes

N/A	Yes	No	
	■	☐	Broken containers/lids?
	■	☐	Missing or incomplete labels?
	■	☐	Illegible information on labels?
	■	☐	Low volume received?
	■	☐	Inappropriate containers received?
☐	■	☐	VOC vials have headspace?
☐	■	☐	Extra sample locations / containers not listed on COC?

Check Sample Preservation

N/A	Yes	No	
	☐	■	Average sample temperature ≤6° C?
☐	☐		Completed Sample Preservation Verification Form?
☐	☐	■	Samples preserved correctly?
☐	☐		If "No", added orange tag?
☐	☐		Received pre-preserved VOC soils? ☐ MeOH ☐ Na_2SO_4

Check for Short Hold-Time Prep/Analyses

☐ Bacteriological
☐ Air Bugs
☐ EnCores / Methanol Pre-Preserved
☐ Formaldehyde/Aldehyde
☐ Green-tagged Containers
☐ Yellow/White-tagged 1L Ambers (SV Prep-Lab)

AFTER HOURS ONLY:
COPIES OF COC TO LAB AREA(S)
☐ NONE RECEIVED
☐ RECEIVED, COCs TO LAB(S)

Notes

☐ Trip blank received ☐ Trip blank not listed on COC
☐ No COC received, Proj. Chemist reviewed (init./date) _____
☐ No analysis requested, Proj. Chemist completed (init./date) _____

Cooler Received (Date/Time)	Paperwork Delivered (Date/Time)	≤1 Hour Goal Met?
		Yes / No

revision: 2.5 | Log In Forms - Receiving.Log-In_Checklist

Approved By: 12-15-08 **QA Officer** | **Approved By:** 12/15/08 **Area Supervisor**

Attachment 6.3
Sample Receiving Log-In Checklist (page 2)

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST - page 2

Project Chemist Use

Notify Laboratory Personnel of Short Hold-Times and/or Rush Work ☐ NONE

(Lab personnel notified/date)

- ☐ Inorganics _____
- ☐ Microbiology (bacteria) _____
- ☐ Metals Prep _____
- ☐ Metals _____
- ☐ GC-Volatiles _____
- ☐ MS-Volatiles _____
- ☐ Semi-Vol. Prep _____
- ☐ GC-Semi-Volatiles _____
- ☐ MS-Semi-Volatiles _____

Log-In Priority ☐ RUSH ☐ Standard

Project Chemist Notes to Log-In Personnel

Trip Blank: ☐ Log-in ☐ Do not log-in

☐ Prep Storage Blank for Client (VOCs)

☐ Sub-Contracting required ☐ Coolant required

☐ Non-TriMatrix or non-standard container type(s) received
Check pH of container type _____
Expected pH:_____ ☐ Adjust if needed

☐ Adjust pH of orange-tagged containers

☐ Lab-filter samples and document on Preservation Form

Sample Narratives to be added at Log-in

Log-In Use

Log Samples into LIMS Sample Nos.

N/A	Yes	
	☐	Receive samples in LIMS
	☐	Date/Time received entered in LIMS match COC
	☐	Read project and submittal narratives
☐	☐	Enter VOC rack/tray number into submittal narrative
	☐	Enter sample information into LIMS
☐	☐	Add any sample narratives
☐	☐	If non-conformance issues, add sample qualifiers
	☐	Print sample number labels

Log-in Analyst (initials/date/time)

Label Sample containers

N/A	Yes	No	
	☐	☐	LIMS label matches tag?
☐	☐		**DISCREPANCIES CORRECTED IN LIMS** Initials/Date: _____
☐	☐		Applicable stickers applied to labels? ☐ MS/MSD sample ☐ Composite before analysis
☐	☐		Applicable stickers applied to containers? ☐ Waste sample ☐ PT sample ☐ USDA regulated
	☐	☐	Orange-tagged containers present?
☐	☐	☐	Adjust pH per Project Chemist
☐	☐		Initials and Date/Time Adjusted on orange tag?
☐	☐		Initials and Date/Time Adjusted on Preservation Form?

Verify Label Accuracy

	☐	Second analyst checked labels for accuracy?
☐	☐	Verify that Orange-tagged containers adjusted/initialed?

Labeled by (initials/date) | Verified by (initials/date)

Sample Storage Check all that apply

bacteria.....	☐ bacteria refrigerator
non-volatiles.....	☐ walk-in cooler
volatiles.....	☐ volatile lab refrigerator
waste.....	☐ waste cabinet
waste VOCs.....	☐ log-in hood refrigerator
low-level Hg	☐ metals lab - DO NOT STORE IN WALK-IN

Paperwork

N/A	Yes	
	☐	original COC (white)
	☐	copy of COC (yellow)
	☐	receiving/log-in checklist
☐	☐	additional cooler information form
☐	☐	sample preservation verification
☐	☐	sample receiving non-conformance form
☐	☐	shipping documents
☐	☐	custody seals
	☐	arrival log
☐	☐	other (note)

For Internal Use Only - DO NOT COPY

revision: 2.5 Log In Forms - Receiving.Log-In_Checklist

Approved By: _____ 12-15-08 **QA Officer** **Approved By:** _____ 12/15/08 **Area Supervisor**

Attachment 6.4
Sample Receiving/Log-In Checklist
Additional Cooler Information

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST
ADDITIONAL COOLER INFORMATION

Recorded by (initials/date)	Client	Project-Submittal No.
	Receipt Log No. / Sample Nos.	Project Chemist

Each cooler block (Cooler No. / Time) records:

Custody Seals
- ☐ none
- ☐ present / intact
- ☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant/Temperature Taken Via:
- ☐ loose ice / avg 2-3 containers
- ☐ bagged ice / avg 2-3 containers
- ☐ blue ice / avg 2-3 containers
- ☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
- ☐ temperature blank (tb)
- ☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

- ☐ Cooler ID on COC?
- ☐ VOC trip blank received?

Cooler No.	Time	Cooler No.	Time	Cooler No.	Time	Cooler No.	Time

Comments

revision: 2.0 Log In Forms - Additional_Cooler_Info

Approved By: 12-15-08 **QA Officer**
Approved By: 12/15/08 **Area Supervisor**

Attachment 6.5
Sample Receiving Non-Conformance Report

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING NON-CONFORMANCE REPORT

Client		Project Submittal No.
Receipt Log No.	Completed By (initials/date)	Project Chemist

List non-conformance issues associated with this submittal in the chart below/left. Identify discrepancies between the COC and sample tags in the chart below/right. Add comments as needed. Give to Project Chemist for immediate action.

		Type of Problem											COC					Sample Tag					LIMS Qualifiers entered for this sample
COC ID No.	Line No.	Discrepancy	Missing Container	Broken Container	Label Missing/ Incomplete	Label Illegible	Low Volume	Inappropriate Container	Headspace	Not Listed on COC	Preservation		Sample Field ID	Date Sampled	Time Sampled	Container Type	Qty	Sample Field ID	Date Sampled	Time Sampled	Container Type	Qty	

Comments:

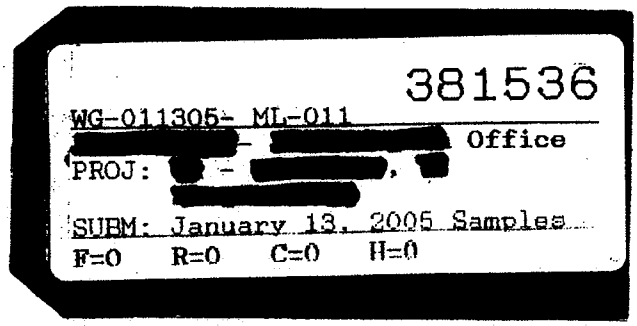
Project Chemist (initials/date):

revision: 1.2 | Log In Forms – Sample_Receipt_Non-Conformance | page ___ of ___

Approved By: 12-15-08
QA Officer

Approved By: 12/15/08
Area Supervisor

Attachment 6.6
TriMatrix Sample Container Tag and Log-In Labels



Project Specific Sample
MS/MSD

TriMatrix Laboratories, Inc.

USDA REGULATED SOIL SAMPLE

Client _____
Project Number: _____
Date: _____ Time: _____
Preservative: H_2SO_4 PINK
Sampled By: _____
Sample Location: _____
_____

Field pH _____
Please verify pH in laboratory.

_____ Initials _____ Date

PE SAMPLE
do not dispose of until notified

Store at Room Temperature

Composite Before Analysis

Composite Before Analysis

Waste Sample
Return to appropriate shelf in waste cabinet

Approved By: _____ 12-15-08 QA Officer
Approved By: _____ 12/15/08 Area Supervisor

Attachment 6.7
Sample Preservation Verification Form

TriMatrix Laboratories, Inc.

SAMPLE PRESERVATION VERIFICATION FORM

page ___ of ___

Client			Project-Submittal No.
Receipt Log No.		Completed By (initials/date)	Project Chemist

COC ID No.			Adjusted by:_____ Date:_____		DO NOT ADJUST pH FOR THESE CONTAINER TYPES			
Container Type	5	4	13		3	6	15	
Tag Color	Lt. Blue	Blue	Brown		Green	Red	Red Stripe	
Preservative	NaOH	H_2SO_4	H_2SO_4		None	HNO_3	HNO_3	
Expected pH	>12	<2	<2		~7	<2	<2	
COC Line No. 1								
COC Line No. 2								
COC Line No. 3								
COC Line No. 4								
COC Line No. 5								
COC Line No. 6								
COC Line No. 7								
COC Line No. 8								
COC Line No. 9								
COC Line No. 10								
Comments								

COC ID No.			Adjusted by:_____ Date:_____		DO NOT ADJUST pH FOR THESE CONTAINER TYPES			
Container Type	5	4	13		3	6	15	
Tag Color	Lt. Blue	Blue	Brown		Green	Red	Red Stripe	
Preservative	NaOH	H_2SO_4	H_2SO_4		None	HNO_3	HNO_3	
Expected pH	>12	<2	<2		~7	<2	<2	
COC Line No. 1								
COC Line No. 2								
COC Line No. 3								
COC Line No. 4								
COC Line No. 5								
COC Line No. 6								
COC Line No. 7								
COC Line No. 8								
COC Line No. 9								
COC Line No. 10								
Comments								

pH strip lot No.
☐ OC481846
☐ _____

Aqueous Samples: For each sample and container type, check the box if pH is acceptable. **If pH is not acceptable for any sample container, record pH in box, and note on Sample Receiving Checklist and on Sample Receiving Non-Conformance Form.** If approved by Project Chemist, add acid or base to the sample to achieve the correct pH. Add up to, but do not exceed 2x the volume initially added at container prep (see table below for initial volumes used). **Add orange pH tag to sample container and record information requested. Record adjusted pH on this form. Do not adjust pH for container types 3, 6, and 15.**

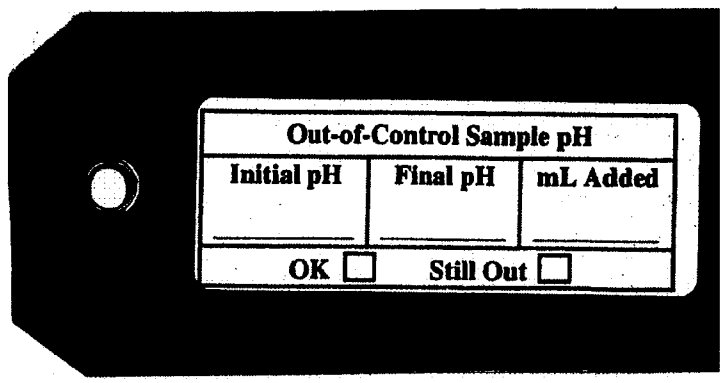
Container Size (mL)	Original Vol. of Preservative (mL)
Container Type 5:	NaOH
500	2.5
1000	5.0
Container Type 4:	H_2SO_4
125	0.5
250	1.0
500	2.0
1000	4.0
Container Type 13	H_2SO_4
500	2.5

version: 1.5 Log In Forms -- Sample_Preservation_Checklist

Approved By: _____ 12-15-08 **QA Officer**

Approved By: _____ 12/15/08 **Area Supervisor**

Attachment 6.8
Out-of-Control Sample pH Tag



Approved By: QA 12-15-08 **Approved By:** 12/15/08
QA Officer **Area Supervisor**

Attachment 6.9
Short Hold Time Analysis List

Short Hold Time Analysis List

Parameter	Hold Time (From Date/Time Sampled)
Inorganics	
Dissolved Oxygen	Field or ASAP
Conductivity	Field or ASAP
Ferrous Iron	Field or ASAP
pH	Field or ASAP
Residual Chlorine	Field or ASAP
Sulfite	Field or ASAP
Coliform, Total and Fecal - Potable Water	30 Hours
Coliform, Total and Fecal - Wastewater	6 Hours
Coliform, Total and Fecal - Non-Potable Water for Compliance Purposes	8 Hours
Coliform, Total and Fecal - Non-Potable Water for Non-Compliance Purposes	24 Hours
Heterotrophic Plate Count - Potable Water	8 Hours
Heterotrophic Plate Count - Non-Potable Water for Compliance Purposes	8 Hours
Heterotrophic Plate Count - Non-Potable Water for Non-Compliance Purposes	24 Hours
Chromium, Hexavalent	24 Hours
BOD	48 Hours
Color	48 Hours
MBAs / Foaming Agents	48 Hours
Nitrogen, Nitrate	48 Hours
Nitrogen, Nitrite	48 Hours
Phosphorus, Ortho	48 Hours
Phosphorus, Dissolved	48 Hours
Turbidity	48 Hours
Organics Prep Lab	
Aldehydes/Formaldehyde - Water	3 Days to extract
Aldehydes/Formaldehyde - Soil	14 Days to Leach, 3 Days after leach to Analyze
DRO - Water 8015	7 Days
DRO - Soil 8015	14 Days
Wisconsin DRO - Water	7 Days
Wisconsin DRO - Soil	10 Days
Semi-Volatiles (BNA/PNA/BN or AN) - Water 8270	7 Days
Semi-Volatiles (BNA/PNA/BN or AN) - Soil 8270	14 Days
PCBs - Water 8082	7 Days
PCBs - Soil 8082	14 Days
Pesticides - Water 8081	7 Days
Pesticides - Soil 8081	14 Days
Herbicides - Water 8151	7 Days
Herbicides - Soil 8151	14 Days
HPLC PNAs - Water	7 Days
HPLC PNAs - Soil	14 Days
Organics in Waste-PCB/Pesticides	14 Days
Organics in Waste-Herbicide	14 Days
Organics in Waste-BNA/PNA/BN or AN	14 Days
Volatiles	
EnCore	48 Hours to Pretreat, 14 Days to Analyze
MeOH Preserved Soil	48 Hours to Pretreat, 14 Days to Analyze
Unpreserved Styrene - Water	7 Days
Unpreserved 2-Chloroethyl Vinyl Ether - Water	7 Days

Short Hold List Revision 2.1

Approved By: 12-15-08 **QA Officer** **Approved By:** 12/15/08 **Area Supervisor**

Attachment 6.10
Container Packing List (page 1)

TriMatrix Laboratories, Inc.

Container Packing List

For any questions regarding these containers, contact a Project Chemist at (616) 975-4500.

Client: Project: Page 1 of 1

#	Sets	Sample Locations	Sample Container Types and Quantities Requested																									
			0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26
1																													
2																													
3																													
4																													
5																													
6																													
7																													
8																													
9																													
10																													
11																													
12																													
13																													
14																													
15																													
16																													
17																													
18																													
19																													
20																													
		Total Containers																											

This container type requires field-filtering

MATRIX	#	TEST	SIZE (mL) / TYPE CONTAINER	OPTIONS	PRESERVATIVE	TAG COLOR
WATER	0	Unpreserved Purgeable Organics	40 mL Clear Glass Vial	40	Cool to 4° C	Yellow & Black Stripe
	1	Preserved Purgeable Organics	40 mL Clear Glass Vial (pre-preserved)	40	HCl; Cool to 4° C	Yellow
	2	Non-Purgeable Organics	1000 mL Amber Glass	1000	Cool to 4° C	Salmon
	3	General Short Hold	Plastic	125, 250, 500, 1000	Cool to 4° C	Green
	4	Nutrients	Plastic	125, 250, 500, 1000	pH <2 w/ H_2SO_4	Dark Blue
	5	Cyanides	500 mL Amber Plastic	500	pH >12 w/ NaOH	Light Blue
	6	Total Metals	Plastic	125, 250, 500, 1000	pH <2 w/ HNO_3	Red
	7	Oil & Grease/TPH	Clear Glass	1000WM, 1000SM	pH <2 w/ H_2SO_4	Dark Blue
	8	Bacteria	125 mL Plastic (pre-preserved)	125	$Na_2S_2O_3$; Cool to 4° C	Pre-Labeled (White)
	9	Sulfide	500 mL Amber Glass + NaOH ampule	500	Zinc Acetate at Lab; NaOH in Field	Light Green
	10	TOX	250 mL Amber Glass w/ Septa Lid	250	pH <2 w/ H_2SO_4	Lilac
	11	TOC	40 mL Amber Vial	40	pH <2 w/ H_2SO_4	Pink
	12	DRO	1000 mL Amber Glass	1000	pH <2 w/ HCl	Gray
	13	Phenols	500 mL Amber Glass	500	pH <2 w/ H_2SO_4	Brown
	14	Formaldehyde	250 mL Amber Glass	250	Cool to 4° C	Orange
	15	Dissolved Metals	Plastic	125, 250, 500, 1000	pH <2 w/ HNO_3	Red & White Stripe
SOIL	16	Inorganics/Metals	WM Plastic	125, 250, 500, 1000	Cool to 4° C	White
	17	Non Purgeable Organics	WM Clear Glass	125, 250, 500, 1000	Cool to 4° C	Manila
	18	Purgeable Organics - Bulk	60 mL WM Clear Glass	60	Cool to 4° C	Light Yellow
	19	TCLP Volatiles	125 mL Clear Glass Vial	125	Cool to 4° C	Yellow & Black Stripe
	20	% Solids	125 mL WM Plastic	125	Cool to 4° C	Yellow & White Stripe
	21	Purgeable Organics	Encore Sampler	5g, 25g	Cool to 4° C	Label on Bag
	22	Purgeable Organics - PrePres.	40 mL Pre-Tared Clear Glass Vial + 10 mL MeOH ampule	40	MeOH in field; Cool to 4° C	Pre-Labeled (Light Yellow added at Lab)
MISC	23					
	24					
	25	Pesticide WWs by Method 608	1000 mL Amber Glass	1000	pH 5-9; Cool to 4° C	Yellow & White Stripe
	26	Drinking Water Volatiles	40 mL Clear Glass Vial	40	Ascorbic Acid at Lab; HCl in Field	Yellow

Notes:

DI Water for Equipment Blanks	Container Type and Size	Qty.
VOC Free		
Millipore		
ASTM Metals Free		

Container Packing List New revision 4.4

Approved By: 12-15-08 **QA Officer** **Approved By:** 12/15/08 **Area Supervisor**

Attachment 6.10
Container Packing List (page 2)

TriMatrix Laboratories, Inc.

Project Chemist Initials	Added to Calendar & Folders (initials/date)	Revision:	Revised By/Date:

Client: _____ Project Manager: _____

Project: _____ Contact: _____

TriMatrix Project No: _____ Date of Request: _____

Type of Order: ○ One-Time ⟹ Due to Client: _____ ○ AM ◉ PM

or

◉ Calendar ⟹ Frequency: ○ Weekly ○ Semi-Annually ○ Monthly ○ Annually ○ Quarterly ◉ Daily

Prepare Containers For:

Months	☐ Jan	☐ Feb	☐ Mar	☐ Apr	☐ May	☐ Jun
	☐ Jul	☐ Aug	☐ Sep	☐ Oct	☐ Nov	☐ Dec
Weeks	☐ 1	☐ 2	☐ 3	☐ 4	☐ 5	
Days	☐ M	☐ T	☐ W	☐ TH	☐ F	

Containers will be ◉ Picked Up or ○ Shipped via: ○ First Overnight ◉ Standard Overnight

Pick up/Ship Date: _____ ○ Priority Overnight ○ Express Saver

Ship Containers to: ○ 2-Day ○ Ground

_____ ○ Saturday Delivery ○ TriMatrix Courier

_____ ○ Other: _____

_____

☐ Shipment to be billed to FedEx Account No.:

Telephone No: _____

Shipment to include: ☐ COCs (Qty) ____ ☐ Custody Seals ☐ Temperature Blanks

☐ MSDS Sheets for all preservatives used ☐ WB TM#? ○ Y ◉ N

Comments: ☐ Cooler Banding Required

_____

_____

_____

Assembled by/Date:	Checked by/Date:	Shipped by/Date:

Cooler Number(s) Used:	Coolers Sealed With Tape	Coolers Sealed With Banding Strap	Tracking Number Label(s):
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	

TriMatrix Laboratories, Inc. 5560 Corporate Exchange Court, Grand Rapids, MI 49512 (616)975-4500

Container Packing List New revision 4.4

Approved By: _____ 12-15-08 **Approved By:** _____ 12/15/08

QA Officer **Area Supervisor**

Attachment 6.11
Waste Cabinet Sample Inventory

TriMatrix Laboratories, Inc.

Waste Cabinet Sample Inventory

Waste Shelf ____

Client Name:	Sample #('s)	Disposal Method	Date

Approved By: TM 12-15-08 **QA Officer** **Approved By:** [signature] 12/15/08 **Area Supervisor**

Attachment 6.12
pH Strip Calibration Logbook

TriMatrix Laboratories, Inc.

pH Strip Calibration Logbook

Date	Lot #	pH 4	pH 7	pH 10	Area

pH STRIP CALIBRATION CRITERIA CORRECTIVE ACTION

1. The acceptance range for the strips is to read the exact pH of the buffer being checked. The wide range strips must pass this criteria at all three levels, 4, 7, and 10. The narrow range pH 5-7 strips are only checked at a pH of 7.

2. If the pH strips do NOT read at their appropriate levels, that lot number must NOT be used. Return them to purchasing.

Approved By: QA 12-15-08 **QA Officer**

Approved By: 12/15/08 **Area Supervisor**

Attachment 6.13
Infra-Red Thermometer Calibration Record

TriMatrix Laboratories, Inc.

INFRA-RED THERMOMETER DECEMBER CALIBRATION RECORD

Date	Time	Initials	NIST Thermometer Reading*	IR Gun Reading	Difference	Acceptable? (<0.50°C Difference)	Comments
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	

*Calibrated vs. Thermometer # (<0.50°C difference at 4° C vs. NIST Thermometer #97 (serial # 1740)

file: IRTHERM page: 12 of 12 revision: 1.0

Approved By: QA 12-15-08
QA Officer

Approved By: 12/15/08
Area Supervisor

Attachment 6.14
Controlled-Temperature Unit Daily Log Sheet

TriMatrix Laboratories, Inc.

Controlled Temperature Unit #32
Daily Log Sheet

Description: Precision Economy Incubator
Model Number: 31750
Serial #: 699091394
Location: Inorganic Laboratory
Purpose: Incubation of Bacteriological Sample Plates
Control Windows: Low: 34.5° C High: 35.5° C
Thermometer #'s: 37, 36
Thermometer Serial #'s: 6187, 6171

Date	Initials	Time of First Readings	First Reading (°C): Top Thermometer	First Reading (°C): Bottom Thermometer	Initials	Time of Second Readings	Second Reading (°C): Top Thermometer	Second Reading (°C): Bottom Thermometer	Adjustments/Observations/Comments

file: NEWTEMP10 page: 14 of 27 revision: 2.2

Approved By: 12-15-08
QA Officer

Approved By: 12/15/08
Area Supervisor

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Bottle Order Preparation

APPROVALS:

Area Supervisor: _____ Date: 5/22/09
Gary L. Wood

QA Officer: _____ Date: 5-22-09
Tom C. Boocher

Operations Manager: _____ Date: 5/26/09
Jeff P. Glaser

Procedure Number: GR-15-101
Revision Number: 0.2

Date Initiated: 3/29/04
Effective Date: 6/1/09

Date Revised: 5/8/09
Pages Revised: All

By: Douglas E. Kriscunas

Total Number of Pages: 25

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 The TriMatrix Laboratories, Inc. policy is to provide sampling kits for all laboratory clients.

1.2 TriMatrix utilizes EPA-approved preservation techniques. Sample kits include pre-cleaned certified sample bottles, certified preservatives, coolers, chain-of-custody forms, custody seals and material safety data sheets.

1.3 Bottle preparation technicians are responsible for performing all tasks applicable to the bottle preparation area, including but not necessarily limited to bottle order preparation and sample container inventory.

1.4 The bottle preparation area supervisor is responsible for annual review of this procedure and technician compliance.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Code of Federal Regulations*, Title 40, Protection of the Environment, Chapter I Environmental Protection Agency, Part 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants, subpart e, Table II, Required Containers, Preservation Techniques, and Holding Times

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, December 1996

3.0 SUMMARY OF PROCEDURE

3.1 Bottle orders are filled as needed and shipped to the client location for sample collection.

3.2 Each bottle includes the appropriate amount of preservative associated with the required analysis.

3.3 Shipping of bottle orders from the laboratory is in a cooler to maintain bottle integrity during transport and sample integrity when samples are returned to the laboratory.

4.0 PARAMETER OR COMPOUND LIST

4.1 There are no parameter and/or compounds directly associated with this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-10-111, *Fixed and Adjustable Mechanical Pipette and Bottle-Top Dispenser Calibration Verification, latest revision*

Approved By: _____ 5-22-09 **Approved By:** _____ 5/15/09
QA Officer **Area Supervisor**

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Contamination must be minimized by the exclusive use of reagent or ACS grade preservatives.

7.0 SAFETY PRECAUTIONS

7.1 Bottle order technicians must comply with all applicable safety instructions as listed in the TriMatrix Safety Manual and Chemical Hygiene Plan.

7.2 A laboratory coat, disposable gloves and approved safety glasses must be worn while working in the bottle order area except when sitting at a computer terminal inputting data.

7.3 No food or drink is permitted in the bottle order area including chewing gum, candy, soft drinks and coffee.

7.4 Pay special attention to the following:

7.4.1 Many sample preservatives are corrosive to the skin, eyes and clothing. Take care to avoid spilling, splashing or handling without disposable gloves. If a minor spill occurs clean it up immediately.

7.4.2 Maintain a clean and uncluttered work surface and area.

7.4.3 The preparation of preservative solutions must be performed in a laboratory hood. Remember to always add concentrated acids to water. **DO NOT** add water to a concentrated acid.

7.5 Material safety data sheets (MSDS) for all chemicals used in the bottle preparation area are stored on the TriMatrix laboratory intranet library.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Specific guidelines for sample collection, preservation, packaging and return shipment are presented in detail as attachments to this procedure.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Micropipette, 100 – 1000 µL, Wheaton Model 822.100 or equivalent

9.2 Bottle-top dispensers, 1 – 5 mL, Brinkman or equivalent

9.3 Macropipette, 1 – 10 mL

9.4 Bubble wrap, bubble bags

9.5 Coolers, various sizes

9.6 Sample containers, various sizes and makes, purchased pre-cleaned to EPA standards.

Note: Sample containers must come with a certificate of cleanness unless otherwise specified. Give these certificates to QA department staff for scanning to the laboratory intranet library. A certificate of lot analysis is available by request through the project chemist.

9.7 Mechanical banding apparatus or approved shipping tape

9.8 Filter paper – Fisher Cat# 09-790-12F or equivalent

9.9 Vacuum-driven, disposable filtration system, Millipore Stericup™, 150 or 250 mL

9.10 Tags, stickers, labels, custom-designed for the laboratory

9.11 Funnels, disposable, polyethylene

9.12 Cotton Gauze Pads – Fisher Cat# 22-415-468 or equivalent

9.13 Adjustable Soil Sampling Plunger (straight type), QEC 0535-0008 or equivalent

9.14 10 gram Soil Sampling T-Handle Device, QEC 0535-0003 or equivalent

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Micropipettes, macropipettes and bottle-top dispensers must be maintained in accordance with TriMatrix SOP GR-10-111.

11.0 CHEMICALS AND REAGENTS

11.1 Nitric acid (HNO_3), trace metals grade

11.2 Sulfuric acid (H_2SO_4), trace metals grade

11.3 Sodium hydroxide (NaOH), ACS grade

11.4 Hydrochloric acid (HCl), trace metals grade

11.5 Sodium thiosulfate pentahydrate ($Na_2S_2O_3[H_2O]_5$) , ACS grade or equivalent

11.6 Zinc acetate dihydrate ($Zn(C_2H_3O_2)_2[H_2O]_2$), ACS grade or equivalent

11.7 EDTA (disodium salt), ACS grade or equivalent

11.8 Hexanes, pesticides grade

11.9 Laboratory reagent water, MilliQ system

11.10 Lead carbonate, ACS grade or equivalent

11.11 Ascorbic acid, ACS grade or equivalent

11.12 Aluminum Chloride ($AlCl_3 \cdot 6H_2O$)

11.13 Methanol – 10 mL PTFE tube, O2SI 19001-02b or equivalent

12.0 STANDARDS PREPARATION

12.1 Nitric acid (50% v/v) – Dilute concentrated HNO_3 with an equal volume of laboratory grade reagent water. Always add the acid to water. Never dilute by adding water to a concentrated acid.

12.2 Sulfuric acid (50% v/v) – Dilute H_2SO_4 with an equal volume of laboratory grade reagent water. Always add the acid to water. Never dilute by adding water to a concentrated acid.

12.3 Sodium hydroxide 10N (50% w/v) – Dilute 500 g NaOH to 1000 mL with laboratory grade reagent water. Dissolve sodium hydroxide carefully as a great amount of heat is given off.

12.4 Sodium hydroxide 6N (24% w/v) – Dilute 240 g NaOH to 1000 mL with laboratory grade reagent water. Dissolve sodium hydroxide carefully as a great amount of heat is given off.

12.5 Hydrochloric acid (50% v/v) – Dilute HCl with an equal volume of laboratory grade reagent water. Always add the acid to water. Never dilute by adding water to a concentrated acid.

12.6 EDTA reagent – Dissolve 2.5 g EDTA disodium salt in 100 mL laboratory grade reagent water.

12.7 Zinc acetate solution – Dissolve 220 g $Zn(C_2H_3O_2)_2[H_2O]_2$ in 870 mL laboratory grade reagent water.

12.8 Aluminum Chloride Solution – Dissolved contents of an unopened 100g bottle into 144mls of Milli-Q reagent water.

12.9 Prepare hexane wipe test jars as follows:

12.9.1 Add no more than 30 mL of hexane to a 120 mL amber wide mouth glass jar.

12.9.2 Place one 3 inch gauze square (wipe) in the jar.

12.9.3 Tighten the screw-cap lid very securely.

12.9.4 Repeat, for as many wipe tests ordered.

12.9.5 Prepare one additional jar and mark as a trip blank. A wipe test trip blank must be prepared for each set of wipe test jars ordered and sent.

12.10 Prepare volatiles trip blanks with organic-free laboratory reagent water in 40 mL VOA vials as follows:

12.10.1 Obtain VOC free reagent from the volatile organic laboratory. Pour laboratory reagent water into a vial to just before over-flowing then tightly cap without headspace. The vial is purchased with hydrochloric acid preservative. If bubbles are present in a filled vial after capping, discard it and remake the blank. A trip blank must be included with each volatiles bottle order.

12.10.2 For potable water trip blanks, use vials with ascorbic acid preservative. Add approximately 20 mL of water and swirl slightly to dissolve the acid. When the acid dissolves, add 2 drops of 1:1 (v/v) hydrochloric acid then fill to just before overflowing with water then tightly cap.

12.10.3 If bubbles are present in a filled vial after capping, discard it and remake the blank.

12.10.4 Each volatiles trip blank vial requires a yellow tag (with trip blank sticker) for recording client, project number, date, time, preservative, sampled by and sample location.

12.10.5 Put prepared volatiles trip blanks in a bubble bag and seal.

12.11 Prepare trip blanks for other analysis requests by filling the appropriate sample container with laboratory reagent water (Milli-Q).

12.12 Prepare rinsate, equipment, atmospheric or other field blanks as follows:

12.12.1 When field blanks are requested, provide more than enough laboratory reagent water to fill all field blank sample containers. Send in a well-sealed container.

12.12.2 Ship the field blank water and sample containers in the same cooler when possible.

12.13 Add preservative to sample containers in accordance to the guideline presented in the Aqueous Preservatives/Volumes Reference Table (refer to Attachment 20.1).

12.14 Fill available cyanide kits as follows and as summarized in the *Available Cyanide Kit Instructions* sheet presented in section 20 of this procedure.

12.14.1 Prepare three 250 mL amber glass bottles as follows:

12.14.1.1 Add 0.25 g lead carbonate to one of the bottles. Attach a "lead carbonate" sticker to the bottle then place in a bubble bag with no tag.

12.14.1.2 Add 1.3 mL 10N sodium hydroxide to the second bottle. Attach a "lead carbonate/sodium hydroxide" sticker and a light blue tag then place in a bubble bag.

12.14.1.3 To the third bottle, add 1.3 mL 10N sodium hydroxide. Attach a "sodium hydroxide" sticker and a light blue tag then place in a bubble bag.

12.14.1.4 Enclose a copy of the TriMatrix Available Cyanide sample collection instruction sheet (refer to Attachment 22.5).

12.14.2 Include the following in each available cyanide kit:

12.14.2.1 A clean disposable polyethylene funnel

12.14.2.2 Filter paper – Fisher Cat# 09-790-12F or equivalent

12.14.2.3 Vacuum-driven disposable filtration system, Stericup™, Millipore, 150 or 250 mL

13.0 ANALYTICAL PROCEDURE

13.1 When a bottle order is received from the project management group:

13.1.1 Review it thoroughly.

13.1.2 Look for special notes or other instructions such as "no acid in metals bottles".

13.1.3 Check all due dates.

13.1.4 Identify whether the order will be for a one-time sampling event or is part of a monitoring project. If part of a monitoring project, place copies of the order in appropriate months in the monthly file cabinet to serve as a reminder that further bottle orders will need prepared. Include the sampling date and site location with each copy filed.

13.2 Prepare a bottle order as follows:

13.2.1 Locate all containers needed to fill the order.

13.2.2 Place preservative in each container, as required.

13.2.3 Label each container, as required.

13.2.4 Tag each container, as required. Each bottle type requires a different colored tag. If a bottle has preservative or a sample is to be preserved, the attached tag must identify the preservative used.

13.2.5 Wrap all glass containers with bubble wrap and pack tightly to prevent breakage during transport.

13.2.6 Be sure to tighten each container lid before wrapping to prevent leaks of preservative.

13.2.7 Select a cleaned, decontaminated cooler(s) for each bottle order. If a cooler requires maintenance, clean according to the following guidelines:

13.2.7.1 Dispose of all loose material from inside the cooler. Place any broken glass in the broken glass receptacle.

13.2.7.2 If there are any unused sample containers with preservative that have been returned unused, they must be rinsed out before putting in the trash. In

particular, when a large number of containers are returned, check with the project chemist on how to proceed. Sometimes client might be billed or bottles saved till their next sampling event.

13.2.7.3 If any container is returned with contents unknown, then seek out area manager or safety officer for further instructions on how to dispose of it. It might be necessary to dispose of contents through waste disposal technician.

13.2.7.4 Scrub outside of the cooler using lab grade soap, tap water and a sponge.

13.2.7.5 Rinse the cooler thoroughly with tap water and dry with paper towels.

13.2.7.6 Inspect cooler for any integrity problems such as punctures, cracks or stains. Tape up any drain plug if not permanently sealed.

13.2.8 Place all site-specific containers together in one cooler, if possible. TriMatrix coolers are numbered/bar coded for monitoring and traceability.

13.2.9 Include chain-of-custody (CoC) forms, material safety data sheets (MSDS) for each preservative used, return shipping labels, a copy of the bottle order form, project notes and additional required items in the cooler. There are a maximum of ten lines to record site locations on the CoC form. Always send enough forms to cover all site locations listed in the bottle order. For example, a bottle order with twelve site locations requires sending at least two forms. If requested, send additional CoC forms.

13.2.10 With bottles and paperwork in the cooler, secure with shipping straps or approved shipping tape.

13.2.11 Record the client name, cooler number, project chemist and tracking number on the second copy of the bottle order. This copy stays at the laboratory.

13.2.12 For clients, ship coolers by United Postal Service (UPS), Federal Express (Fedex) or other carrier. For TriMatrix field service sampling, place in the laboratory area required.

13.3 After completing a bottle order, notify the project chemist to perform an inspection and sign-off of the order. Do not send a bottle order without project chemist authorization.

13.4 Following inspection and sign-off from a project chemist, tape or band the cooler tightly, add a custody seal of requested, placard with "Package Conforms to 49 CFR 173.4" if any sample container contains chemical preservatives and ship in accordance to method prescribed on the bottle order.

14.0 DATA REPORTING ANDS DELIVERABLES

14.1 Perform daily calibration check on all reagent dispensers at 1.3 and 2.5 mLs by dispensing into a 10 mL glass graduated cylinder. Record results of calibrations checks in the *Calibration Verification Logbook.*

14.2 Record the current date prepared and lot number of all reagents in the *Reagent Use Logbook.*

14.3 Complete page two of all bottle orders / packing list with the appropriate information as related to sample kit preparation, packaging and shipment.

14.4 Complete any additional paperwork as needed.

15.0 QUALITY ASSURANCE

15.1 General workroom hygiene and reminders:

15.1.1 Remember to add the appropriate color-coded label to all preserved sample containers.

15.1.2 Decontaminate coolers as required or scheduled.

15.1.3 Clean counters between orders and when necessary to maintain chemical hygiene.

15.1.4 Dust shelves at least weekly to prevent dust and contamination build-up.

15.1.5 Tear down cardboard to place in the recycling bin at the end of each day or when necessary to maintain a safe working environment.

15.1.6 Make sure paper is in the appropriate bin to be shredded.

15.1.7 No food and/or drink are allowed in the bottle preparation area. Safety glasses must be worn.

15.2 Bottle order technicians must review each completed section of the bottle order form for accuracy and completion before submitting to the project chemist. If a there is a discrepancy or question that can not be resolved, consult with the project chemist before continuing with the order.

15.3 When a bottle order has been completed and before sending to the client, the project chemist must review, verify and confirm the accuracy and completion of the order. Only with project chemist approval can a bottle order be shipped.

16.0 DEMONSTRATION OF CAPABILITIES

16.1 New technicians must be trained in this procedure during orientation and with each updated revision.

16.2 Document training by completing and signing training forms which can be obtained from the Quality Assurance department.

17.0 POLLUTION PREVENTION

17.1 Maintain an adequate inventory of all chemicals used in the bottle preparation area and monitor their use by completing the appropriate logbooks referenced in the procedure.

Approved By: _____ 5-22-09 **QA Officer** **Approved By:** _____ 5/15/09 **Area Supervisor**

17.2 Never dispose of chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranet library in the MSDS and CoA folders.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 (Laboratory Waste Disposal) for laboratory waste disposal.

19.0 REFERENCES

19.1 *Code of Federal Regulations*, Title 40, Protection of the Environment, Chapter I Environmental Protection Agency, Part 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants, subpart e, Table II, Required Containers, Preservation Techniques, and Holding Times

19.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, December 1996

20.0 ATTACHMENTS

20.1 TriMatrix Aqueous Preservatives/Volumes Reference Table

20.2 Example Container Packing List - Container Detail

20.3 Example Container Packing List – Frequency/Shipping/Quality Control Detail

20.4 Sample Container Ordering Information

20.5 Available Cyanide Kit Instructions (kit preparation)

20.6 Available Cyanide Collection Instructions

20.7 Total Sulfide Sample Collection Instructions

20.8 Dissolved Sulfide Sample Collection Instructions

20.9 Drinking Water Volatile Organic Collection Instructions

20.10 Low-Level Mercury (LLHg) Sample Kit Information

20.11 Methanol (MeOH) Field Preservation Kit Information

20.12 Sample Collection, Packaging and Return Shipment Guide

20.13 Example – Commercial Invoice for International Shipments

20.14 Example – Instruction Sheet for USDA Regulated Soil Materials

Attachment 20.1
TriMatrix Aqueous Preservatives/Volumes Reference Table (Revision 21)

#	DESCRIPTION	TAG COLOR	PRESERVATIVE REQUIREMENT	QUANTITY INITIALLY ADDED (mL)
0	40 mL Vial - Unpreserved Purgeable Organics	Yellow/Black Stripe	Cool to 4° C	-----
1	40 mL Vial - Preserved Purgeable Organics	Yellow	pH <2 w/ 1:1 HCl, Cool to 4° C	Purchased Pre-Preserved
2	1000 mL Amber Glass Non Purgeable Organics	Salmon	Cool to 4° C	-----
3	125 250 500 1000 mL Plastic - Non Preserved	Green	Cool to 4° C	-----
4	125 mL Plastic - Nutrients	Dark Blue	pH <2 w/ H_2SO_4	0.5
4	250 mL Plastic - Nutrients	Dark Blue	pH <2 w/ H_2SO_4	1.0
4	500 mL Plastic - Nutrients	Dark Blue	pH <2 w/ H_2SO_4	2.0
4	1000 mL Plastic - Nutrients	Dark Blue	pH <2 w/ H_2SO_4	4.0
5	500 mL Amber Plastic - Cyanides	Light Blue	pH >12 w/ NaOH	2.5
5	1000 mL Amber Plastic - Cyanides	Light Blue	pH >12 w/ NaOH	5.0
6	125 mL Plastic - Total Metals	Red	pH <2 w/ HNO_3	0.6
6	250 mL Plastic - Total Metals	Red	pH <2 w/ HNO_3	1.3
6	500 mL Plastic - Total Metals	Red	pH <2 w/ HNO_3	2.5
6	1000 mL Plastic - Total Metals	Red	pH <2 w/ HNO_3	5
7	1000 mL Glass - Oil & Grease/TPH / WM NM	Dark Blue	pH <2 w/ H_2SO_4	5
8	120 mL Plastic Bacteria	White Label	Cool to 4° C	Purchased w/Sodium Thiosulfate Tablet
9	500 mL Amber Glass - Total Sulfide	Light Green	Zinc Acetate and NaOH	1.0 mL 2N Zinc Acetate 2.0 mL 10N NaOH
9	250 mL Amber Glass 40 mL Vials - Dissolved Sulfide	Light Green	NaOH in 250 mL Amber Zinc Acetate and NaOH in Vials Container of Aluminum Chloride	See Dissolved Sulfide Sample Collection Instructions
10	250 mL Amber Glass - TOX	Lilac	pH <2 w/ H_2SO_4	1.3
11	40 mL Amber Glass - TOC	Pink	pH <2 w/ H_2SO_4	Purchased Pre-Preserved
12	1000 mL Amber Glass - DRO	Gray	pH <2 w/ 1:1 HCl	5.0
13	500 mL Amber Glass - Phenols	Brown	pH <2 w/ H_2SO_4	2.5
14	250 mL Amber Glass - Formaldehyde	Orange	Cool to 4° C	-----
15	125 mL Plastic - Dissolved Metals	Red/White Stripe	pH <2 w/ HNO_3	0.6
15	250 mL Plastic - Dissolved Metals	Red/White Stripe	pH <2 w/ HNO_3	1.3
15	500 mL Plastic - Dissolved Metals	Red/White Stripe	pH <2 w/ HNO_3	2.5
15	1000 mL Plastic - Dissolved Metals	Red/White Stripe	pH <2 w/ HNO_3	5
22	500 mL Plastic or Amber Glass - Sulfite	White	5 mL EDTA	5
23	250 mL Plastic - Ferrous Iron	White	Conc. HCl	5

Approved By: _____ 5-22-09
QA Officer

Approved By: _____ 5/15/09
Area Supervisor

Attachment 20.2
Container Packing List – Container Detail

TriMatrix Laboratories, Inc.

Container Packing List

For any questions regarding these containers, contact Jennifer Rice at (616) 940-4277.

Client: [redacted] Project: [redacted] Page 1 of 1

#	Sets	Sample Locations	Sample Container Types and Quantities Requested																								
			0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25
1																												
2	20	Qtrly GW				1	1	1						3				1										
3	1	Qtrly LDS w/ MS/MSD		4		1	1		1																			
4	6	Qtrly LDS		2		1	1		1																			
5	1	Leachate composite		2		1	1		1																			
6	2	S/A Surface Water				3	1		1																			
7																												
8	3	VOC resamples or field blanks		2																								
9																												
10		Trip Blanks		2																								
11																												
12																												
13																												
14		Di Water - See Below																										
15																												
16																												
17																												
18																												
19																												
20																												
		Total Containers		24		34	30	20	10					60				20										
																		This container type requires field-filtering										

MATRIX	#	TEST	SIZE (mL) / TYPE CONTAINER	OPTIONS	PRESERVATIVE	TAG COLOR
WATER	0	Unpreserved Purgeable Organics	40 mL Clear Glass Vial	40	Cool to 4° C	Yellow & Black Stripe
	1	Preserved Purgeable Organics	40 mL Clear Glass Vial (pre-preserved)	40	HCl; Cool to 4° C	Yellow
	2	Non-Purgeable Organics	1000 mL Amber Glass	1000	Cool to 4° C	Salmon
	3	General ☐ Short Hold	1000 mL Plastic	125, 250, 500, 1000	Cool to 4° C	Green
	4	Nutrients	500 mL Plastic	125, 250, 500, 1000	pH <2 w/ H_2SO_4	Dark Blue
	5	Cyanides	500 mL Amber Plastic	500	pH >12 w/ NaOH	Light Blue
	6	Total Metals	500 mL Plastic	125, 250, 500, 1000	pH <2 w/ HNO_3	Red
	7	Oil & Grease/TPH	Clear Glass	1000WM, 1000NM	pH <2 w/ H_2SO_4	Dark Blue
	8	Bacteria	125 mL Plastic (pre-preserved)	125	$Na_2S_2O_3$; Cool to 4° C	Pre-Labeled (White)
	9	Sulfide	500 mL Amber Glass + NaOH ampule	500	Zinc Acetate at Lab; NaOH in Field	Light Green
	10	TOX	250 mL Amber Glass w/ Septa Lid	250	pH <2 w/ H_2SO_4	Lilac
	11	TOC	40 mL Amber Vial	40	pH <2 w/ H_2SO_4	Pink
	12	DRO	1000 mL Amber Glass	1000	pH <2 w/ HCl	Gray
	13	Phenols	500 mL Amber Glass	500	pH <2 w/ H_2SO_4	Brown
	14	Formaldehyde	250 mL Amber Glass	250	Cool to 4° C	Orange
	15	Dissolved Metals	500 mL Plastic	125, 250, 500, 1000	pH <2 w/ HNO_3	Red & White Stripe
SOIL	16	Inorganics/Metals	WM Plastic	125, 250, 500, 1000	Cool to 4° C	White
	17	Non Purgeable Organics	WM Clear Glass	125, 250, 500, 1000	Cool to 4° C	Manila
	18	Purgeable Organics - Bulk	60 mL WM Clear Glass	60	Cool to 4° C	Light Yellow
	19	TCLP Volatiles	125 mL Clear Glass Vial	125	Cool to 4° C	Yellow & Black Stripe
	20	% Solids	125 mL WM Plastic	125	Cool to 4° C	Yellow & White Stripe
	21	Purgeable Organics	Encore Sampler	5g, 25g	Cool to 4° C	Label on Bag
	22	Purgeable Organics - PrePres.	40 mL Pre-Tared Clear Glass Vial + 10mL MeOH ampule	40	MeOH in field; Cool to 4° C	Pre-Labeled (Light Yellow added at Lab)
MISC	23					
	24					
	25	Pesticide WVs by Method 608	1000 mL Amber Glass	1000	pH 5-9; Cool to 4° C	Yellow & White Stripe

Notes:

DI Water for Equipment Blanks	Container Type and Size	Qty.
VOC Free	250 ml amber glass	2
Millipore		
ASTM Metals Free		

[redacted]

revision 4.2

Approved By: [signature] 5-22-09 **QA Officer**

Approved By: [signature] 5/15/09 **Area Supervisor**

Attachment 20.3
Container Packing List – Frequency/Shipping/Quality Control Detail

TriMatrix Laboratories, Inc.

Project Chemist Initals	Added to Calender & Folders (initials/date)	Revision:	Revised By/Date:

Client: [redacted] Project Manager:

Project: [redacted] Contact:

TriMatrix Project No: **35525** Date of Request: 11/7/2007

Type of Order: ○ One-Time ⟹ Due to Client: **Nov 12, 2007** ○ AM ◉ PM

or

◉ Calendar ⟹ Frequency: ○ Weekly ○ Semi-Annually ○ Monthly ○ Annually ○ Quarterly ◉ _____

Prepare Containers For:

Months	☐ Jan	☐ Feb	☐ Mar	☐ Apr	☐ May	☐ Jun
	☐ Jul	☐ Aug	☐ Sep	☐ Oct	☐ Nov	☐ Dec
Weeks	☐ 1	☐ 2	☐ 3	☐ 4	☐ 5	
Days	☐ M	☐ T	☐ W	☐ TH	☐ F	

Containers will be ○ Picked Up or ◉ Shipped via: ○ First Overnight ○ Standard Overnight

Pick up/Ship Date: **Nov 12, 2007** ○ Priority Overnight ○ Express Saver

Ship Containers to: ○ 2-Day ○ Ground

Rob ○ Saturday Delivery ◉ TriMatrix Courier

EnviroSolutions ○ Other: _____

☐ Shipment to be billed to FedEx Account No.:

Telephone No: _____

Shipment to include: ☑ **COCs (Qty)** 7 ☐ Custody Seals ☐ Temperature Blanks

☐ MSDS Sheets for all preservatives used ☐ WB TM#? ○ Y ◉ N

Comments: ☐ Cooler Banding Required

Assembled by/Date:	Checked by/Date:	Shipped by/Date:

Cooler Number(s) Used:	Coolers Sealed With Tape	Coolers Sealed With Banding Strap	Tracking Number Label(s):
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	

TriMatrix Laboratories, Inc. 5560 Corporate Exchange Court, Grand Rapids, MI 49512 (616)975-4500

[redacted] revision 4.2

Approved By: _____ 5-22-09 **QA Officer**

Approved By: _____ 5/15/09 **Area Supervisor**

Attachment 20.4
Sample Container Ordering Form

TriMatrix Laboratories, Inc.

Supplies Order Form

Inventory Completed: Date: Time: Initials:

Size\mL	Description	#/ Case	Target	On Hand	Need	QEC Order #	Other Order #	# Received	Date Received
			Plastics						
125	WM Plastic	500	1r / 2s			6212-0004	16060-008		
250	WM Plastic	250	1r / 2s			6212-0008	16060-010		
500	WM Plastic	160	1r / 5s			6212-0016	16238-162		
500	NM Amber Plastic	152	3			6223-0016	16062-109		
1000	WM Plastic	82	2r /8s			6212-0034			
			Glass Containers						
20	Clear vial	72	5			2112-20ml			
40	HCl Preserved Clear vial	72	25			3112-40HCL			
40	HCl vial (for Metals LLHg)	72	10			3112-40HCL			
40	H2SO4 Preserved Amber vial	72	25			3122-40H2SO4			
40	Tare Weight Vial	72	5			2112-40ml TW			
40	Clear vial	72	6			2112-40ml			
60	Clear Short Glass	144	2			6114-0002	ir220-0060		
60	Amber Tall Glass (Pre-tarred)	24	10			2200060FTW			
120	WM Amber glass	12	10			571952	ir241-0120		
250	NM Amber glass w/ Septa lid	24	3			2121-S008	irs249-0250		
250	NM Amber glass	96	2			6121-0008	ir249-0250		
500	NM Amber glass	60	2			6121-0016	ir249-0500		
1000	NM Amber glass	30	16			2121-0032	ir249-1000		
1000	WM Amber glass	36	1			6122-0032			
1000	NM Clear glass	30	6			2111-0032	ir229-1000		
1000	WM Clear glass	12	14			2114-0032	ir221-1000		
125	Clear glass jar	144	2			6114-0004	ir220-0125		
250	Clear glass jar	24	8			2114-0009	ir220-0250		
500	Clear glass jar	12	15			2114-0016	ir220-0500		
			Miscellaneous						
120	Bacti Bottle w/Sodium Thio.	300	1r / 1s				spb125-2052		
5g	Encores	50	4			0535-0004	en5g		
25g	Encores (minimum 4cases)	50	4			0535-0005	en25g		
2"	Tape for tape gun	48	1			(seal-tech)	C263-48X100C		
7"x7 1/2"	Large Bubble Bags	200	30				BB77511NT		
5"x5 1/2"	Small Bubble Bags	1000	8				BB5551WT		
2'x1'	Bubble Roll	2	2				BW48750-24/12		
10	Methanol tubes	100	1				19001-02b		
10	Plungers - Straight	100	1			0535-0008			
10	Plungers - T Handle	100	1			0535-0003			
Yellow	H2SO4 Stickers	1000	6				ir503-0004		
Red	HNO3 Stickers	1000	6				ir503-0005		
Brown	Zinc Acetate/ NaOH Stickers	1000	2				ir503-0006		
Blue	HCl Stickers	1000	2				ir503-0007		
Pink	NaOH Stickers	1000	6				ir503-0008		
Green	Na2S2O3 Stickers	1000	1						
10x12	Ziplock Bags	1000	2				L3602		
10x4x18	Gussetted Bags	1000	2				64-3-65		
150	Stericups	12	4			SCHVU01RE			
4mL	4 ml Vials	144	2			L4640			
4mL	4 mL Caps	144	2			L4200			
65 mm	Plastic Funnels Short Stem	100	1			L5380			
15 cm	Filter Papers P8-creped	100	2				09-790-12F		

Inventory Ordered: Date: Time: Initials:

Approved By: _____ 5-22-09 **QA Officer**

Approved By: _____ 5/15/09 **Area Supervisor**

Attachment 20.5
Available Cyanide Kit Instructions (kit preparation)

TriMatrix Laboratories, Inc.

Available Cyanide Kit Instructions

Each Sample Needs:

(3) 250mL Amber Glass Bottles
(1) 250mL Stericup Presterilized Millipore Filter
(1) Filter Paper
(1) Plastic Filter

- 250mL Amber Glass Bottle
  - White Lead Carbonate Sticker
  - PbCO3 I-Chem Sticker
  - .25g PbCO3
  - No Tag
- 250mL Amber Glass Bottle
  - White Lead Carbonate / Sodium Hydroxide Sticker
  - Pink NaOH Preservative Sticker
  - 1.3mL NaOH
  - Light Blue Tag
- 250mL Amber Glass Bottle
  - White Sodium Hydroxide Sticker
  - Pink NaOH Preservative Sticker
  - 1.3mL NaOH
  - Light Blue Tag

Note: Don't forget to add yellow Available Cyanide Sample Collection packet to paperwork!

*** (1) Instruction packet per 19 kits

Approved By: _____ 5-22-09 **QA Officer**

Approved By: _____ 5/15/09 **Area Supervisor**

Attachment 20.6
Available Cyanide Collection Instructions

TriMatrix Laboratories, Inc.

IMPORTANT INFORMATION FOR AVAILABLE CYANIDE SAMPLE COLLECTION

Two sample containers must be collected at each sample point. One container will be treated with lead carbonate and sodium hydroxide, and the second with only sodium hydroxide (see below and the attached flowchart). A form titled "Available Cyanide Sample Treatment Record" has been provided to document all field pre-treatment activities. Please complete it as you collect and treat each sample. If you have any questions on the treatment procedures described below, please contact your project chemist at 1-616-975-4500.

IMPORTANT: To avoid analyte loss it is required that all sample treatments occur within 15 minutes of sample collection.

CAUTION: All containers labeled as Sodium Hydroxide and Lead Carbonate/Sodium Hydroxide contain 1.3 mL of 10N sodium hydroxide. This solution is very caustic. Avoid skin contact. Handle with care.

CAUTION: All containers labeled as Lead Carbonate contain 0.25 g of solid lead carbonate. Avoid inhalation and skin contact.

1.0 Sample Collection Equipment

Per Sample
- One membrane filter
- One plastic powder funnel
- One sheet of filter paper
- One Lead Carbonate bottle
- One Lead Carbonate/Sodium Hydroxide bottle
- One Sodium Hydroxide bottle

A hand pump (not provided) is also required to perform this procedure

2.0 Collecting a Lead Carbonate/Sodium Hydroxide Pre-Treated Sample

If the sample contains particulates, begin with section 2.1. If the sample is particulate free, begin with section 2.2.

2.1 Sample Contains Particulate Matter

If the sample contains particulate matter that would be removed upon filtration, the sample must be filtered prior to the lead carbonate pre-treatment to avoid the loss of any cyanides associated with the particulate matter. Using a powder funnel and a sheet of filter paper, filter the sample into the bottle labeled Lead Carbonate. Filter enough sample to fill the bottle up to its neck. Place the used filter paper into the bottle labeled Lead Carbonate/Sodium Hydroxide. Cap the Lead Carbonate bottle and gently swirl to mix the sample and the lead carbonate. The sulfide will react with the lead carbonate

tp available cyanide instruct.doc Page 1 of 2 revision: 0.1

TriMatrix Laboratories, Inc.

Available Cyanide Sample Collection

and precipitate out as lead sulfide. The sample must now be filtered through a membrane filter to prevent the loss of any cyanide through reaction with the precipitated lead sulfide. Using a new membrane filter apparatus and a hand pump, filter the sample. Transfer the filtrate into the Lead Carbonate/Sodium Hydroxide bottle containing the used filter paper. Do not pre-rinse the container or fill to overflowing, as a loss of the particulate matter and sodium hydroxide will result. Proceed to section 3.0.

2.2 Sample Particulate Free

With a minimum of aeration, fill the 250 mL bottle labeled Lead Carbonate up to the neck with sample. Cap and gently swirl to mix the sample and the lead carbonate. The sulfide will react with the lead carbonate and precipitate out as lead sulfide. The sample must now be filtered through a membrane filter to prevent the loss of any cyanide through reaction with the precipitated lead sulfide. Using a new membrane filter apparatus and a hand pump, filter the sample. Transfer the filtrate collected into the bottle labeled Lead Carbonate/Sodium Hydroxide. Do not pre-rinse the container or fill to overflowing to avoid the loss of the sodium hydroxide.

3.0 Collecting a Sodium Hydroxide Pre-Treated Sample

With a minimum of aeration fill the 250 mL bottle labeled Sodium Hydroxide with sample. Do not pre-rinse the container or fill to overflowing to avoid the loss of the sodium hydroxide.

4.0 Collect all Paperwork and Return the Samples to TriMatrix

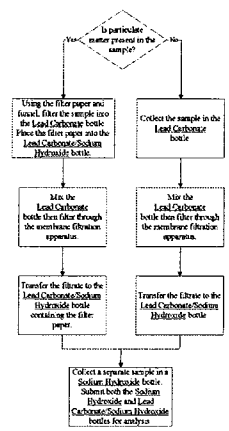
Place all samples in the cooler. Surround the samples with ice. To avoid data qualification all samples must be received at a temperature of between 0 and 6° C. Seal all paperwork in the resealable bag. Place the sealed bag containing the paperwork. Place all plastic powder funnels and unopened membrane filters in the cooler. Seal the cooler and return it to TriMatrix.

If you have any questions, please call TriMatrix at 1-616-975-4500 and speak with your project chemist. Thank you.

bp available cyanide instruct.doc page 2 of 2 revision: 0.2

TriMatrix Laboratories, Inc.

Available Cyanide Sample Collection Flowchart



file: tp available cyanide sample collection flowchart.doc Page: 1 of 1 revision: 0.1

TriMatrix Laboratories, Inc.

Available Cyanide Sample Treatment Record

Sampled By: Company: Date:

	Sample ID	Time Collected	Did Sample Contain Particulate Matter and Require Filtration?	Sample Combined with Lead Carbonate	Filtrate Combined with Sodium Hydroxide	Time Treatment Completed	Treatment Completed within 15 minutes of Collection?	Preserved Sample Collected
1)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
2)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
3)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
4)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
5)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
6)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
7)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
8)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
9)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
10)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
11)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
12)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
13)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
14)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
15)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
16)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
17)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
18)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No
19)			Yes / No	Yes / No	Yes / No		Yes / No	Yes / No

BP Available Cyanide Sample Field Treatment.xls revision: 0.0

Approved By: _____ 5-22-09 **QA Officer**

Approved By: _____ 5/15/09 **Area Supervisor**

Attachment 20.7
Total Sulfide Collection Instructions

TriMatrix Laboratories, Inc.

IMPORTANT INFORMATION FOR SULFIDE SAMPLE COLLECTION

The amber, 500 mL, light green-tagged bottles supplied for sulfide sample collection have been pre-preserved with 1 mL of 2N zinc acetate. Sulfide samples must also be preserved with sodium hydroxide to a pH of ≥9; however, to correctly preserve the sulfide in the sample the addition of the sodium hydroxide must be made *after* the sample has been combined with the zinc acetate. A 4 mL vial containing 2 mL of 10N sodium hydroxide has been included with every 500 mL sulfide sample bottle for this purpose.

With a minimum of aeration, fill a 500 mL bottle up to the neck with sample. Cap and gently swirl to mix the sample and the zinc acetate. Open the sample bottle and transfer all of the sodium hydroxide from one of the 4 mL vials. Carefully add more sample to fill the 500 mL bottle, cap and mix. The filled sample container should be headspace free.

CAUTION: The 10N sodium hydroxide solution is very caustic. Handle with care.

Please call 1-616-975-4500 and speak to your project chemist with any questions. Thank you.

Approved By: _____ 5-22-09 **QA Officer** **Approved By:** _____ 5/15/09 **Area Supervisor**

Attachment 20.8
Dissolved Sulfide Sample Collection Instructions

TriMatrix Laboratories, Inc.

Dissolved Sulfide Sample Collection and Preservation

To measure dissolved sulfide, insoluble matter in the sample must first be removed. This is accomplished by producing an aluminum hydroxide floc using sodium hydroxide and aluminum chloride. The flocculent is allowed to settle and the supernatant decanted off and preserved with zinc acetate. It is important that there is no headspace present in the bottle after the addition of the aluminum chloride. The vials containing the final decanted sample must also be headspace free. If you have any questions on the treatment procedures described below, please contact your project chemist at 1-616-975-4500.

Supplies

Quantity	*Item*
1 per sample	250 mL amber bottle containing 0.5 mL (10 drops) 6N NaOH
2 per sample	40 mL VOA vials, each containing 0.1 mL (2 drops) 2N Zinc Acetate per sample
2 or 3	eye droppers
1	Container of Aluminum Chloride. Enough has been sent to allow for the addition of 10 drops (0.5 mL) to each 250 mL sample.

Procedure

1.0 Collect the sample in the 250 mL amber bottle containing the NaOH. Completely fill the bottle (must be enough sample so when capped it is headspace free).

2.0 Immediately add 10 drops of the Aluminum Chloride solution.

3.0 Mix the sample by holding the bottle in an upright position and rotating your wrist back and forth for 1 minute.

4.0 Allow the sample to settle for 5 to 15 minutes (long enough to allow the flocculent to settle to the bottom of the bottle but not longer than 15 minutes). Wait only as long as necessary to collect 80 mL of supernatant.

5.0 Carefully decant the supernatant into the (2) 40 mL VOA vials containing the 2N zinc acetate. Completely fill the vials with sample so they are headspace free.

6.0 The sample remaining in the 250 mL amber bottle is caustic. Please return the partially filled bottle to TriMatrix for disposal.

Approved By: 5-22-09 **QA Officer** **Approved By:** 5/15/09 **Area Supervisor**

Attachment 20.9
Collection of Volatile Organic Drinking Water Samples

TriMatrix Laboratories, Inc.

IMPORTANT INFORMATION FOR THE COLLECTION OF VOLATILE ORGANIC DRINKING WATER SAMPLES

Open the water tap and allow the system to flush until the water temperature has stabilized (usually about 10 minutes). Reduce the water flow and carefully collect a set of duplicate samples. It is important that the flow is slow enough that no air bubbles pass through the sample as the vial is being filled. Each 40 mL vial has been pre-preserved with 25 mg of ascorbic acid preservative. Fill sample vials to just overflowing, taking care not to flush out the ascorbic acid.

Prior to sealing the set of vials, each sample must also be preserved with 1:1 hydrochloric acid. Using the supplied eyedropper and vial of HCl, carefully add 2 drops of HCl to each vial. The HCl must be added after the collection of the sample. DO NOT add the HCl to the sample vial prior to collecting the sample.

CAUTION: The 1:1 HCl is very acidic. Handle with care.

NOTE: If the sample foams vigorously when the HCl is added, discard that set of samples. Collect a new set, omitting the addition of the HCl. These samples must be flagged as "not acidified" on the chain of custody.

Seal the vials, invert, and mix for 1 minute. Verify that the sealed and mixed vial is bubble and headspace free. Sample data generated from vials received with headspace will be qualified accordingly.

The samples must be chilled to about 4° C when collected, and maintained at that temperature until analysis. Samples must be packaged for shipment with sufficient ice to ensure they arrive at the laboratory with a substantial amount of ice remaining in the cooler. Do not use Blue Ice. Surrounding the samples with crushed or cubed ice is strongly recommended. Samples received at the laboratory within 6 hours of collection may not have sufficient time to cool to 4° C. Provided that they have been correctly packed in ice, no qualifications will be necessary. Samples received in excess of 6 hours of the time of collection that exceed the required preservation temperature will be qualified accordingly.

Please call 1-616-975-4500 and speak to your project chemist if you have any questions. Thank you.

volatile drinking water instructions.doc | Page 1 of 1 | revision: 0.0

Approved By: _____ 5-22-09 **QA Officer**

Approved By: _____ 5/15/09 **Area Supervisor**

Attachment 20.10
Low Level Hg (LLHg) Instructions

TriMatrix Laboratories, Inc.

LOW-LEVEL MERCURY (LLHg) SAMPLE KIT INFORMATION

Each LLHg kit provided with this container shipment consists of the following:

1) Two (2) 40 ml vials containing 0.5ml of concentrated hydrochloric acid (HCL) in reagent water, double bagged in two (2) Ziploc® type storage bags.
2) Sample labels.
3) Shipping-bubble bag.

Sample Collection:

1) In an effort to maintain the purity of each lot of prepared sample vials, the acidified reagent water is to be removed just prior to sample collection.
2) Following the "dirty hands and "clean hands" procedures from USEPA Method 1669, the sample containers are removed from the storage bags.
3) Open each sample vial and empty the acidified reagent storage water into the waste container provided or approved waste stream.
4) The sample container and cap should then be rinsed three times with the sample to be collected.
5) The sample containers should then be completely filled with sample so that when the septum cap is fitted and sealed, and the vial inverted, little or no headspace is visible.
6) Insert vials into the inner storage bag and seal the bag followed by outer bag.
7) Place sample properly labeled vials in shipping bag provided. Samples for total or dissolved mercury do not require refrigeration during sample shipment.
8) Samples must arrive at the laboratory within 48 hours of collection.

Please note:

1) If requested, a waste container has been provided for collection of the acidified reagent water and should be returned to the laboratory for deposal.

2) Specific sample collection procedures are presented in USEPA Methods; 1669 "*Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels July 1996"and* USEPA Method 1631E "*Mercury in Water by Oxidation Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry.*

CAUTION: The sample bottle storage water is acidic. Handle with care.

Please call 1-616-975-4500 and speak to your project chemist with any questions.

llhg instructions .doc | Page 1 of 1 | revision: 1.0

Approved By: _____ 5-22-09 **QA Officer**

Approved By: _____ 5/15/09 **Area Supervisor**

Attachment 20.11
Methanol (MeOH) Field Preservation Kit Instructions

TriMatrix Laboratories, Inc.

METHANOL (MeOH) FIELD PRESERVATION FOR SOIL VOCs SAMPLE KIT INFORMATION

Each MeOH kit provided with this container shipment consists of the following:

1) One (1) tare weighted 40mL vial.
2) One (1) 10mL sealed MeOH tube.
3) One (1) Soil sample collection device, consisting of either a variable straight plunger or a 10 gram T-Handled coring device.
4) Shipping bubble bag

Sample collection

Specific sample collection procedures are presented in USEPA Method 5035A "*Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Draft Revision 1, July, 2002*" and MDEQ Operational Memorandum No. 2, Attachment 6, "*Sampling Methods for Volatile Organic Compounds in Soils, July 5, 2007*" and are summarized as follows:

1) Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Always wear gloves whenever handling the tared sample vials.
2) Using the sample collection device provided, collect approximately 10g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere.
3) Carefully wipe the exterior of the sample collection device with a clean cloth or towel. Using the sample collection device, add about 10g (4 - 6 cm) of soil to the vial.
4) Add the10mL of methanol by cutting open the seal methanol tube and carefully pouring the contents into the sample vial.
5) Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Place sample vial in bubble bag provided and store on ice at 4°C.
6) When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 10.0 ± 0.5g of sample was added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed. Record the weight of the sealed vial containing the sample to the nearest 0.01g. Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 10.0 ± 0.5g. Discard each trial sample.
7) The collection of at least one additional sample is required for the determination of the percent solids (optional container or other container provided for non-volatile analyte testing).

Please call 1-616-975-4500 and speak to your project chemist with any questions.

meoh instructions .doc | Page 1 of 1 | revision: 1.0

Approved By: 5-22-09 **QA Officer** **Approved By:** 5/15/09 **Area Supervisor**

Attachment 20.12
Sample Collection, Packaging and return Shipment Guide

TriMatrix Laboratories, Inc.

Sample Collection, Packing and Return

All supplied containers are pre-cleaned, no additional cleaning is required. Some containers have preservatives present in them. Please do not rinse or overfill. Removal of some or all of the preservative may result in qualified data. Most of the chemicals used as preservatives are hazardous. Use caution when handling. Do not breathe or come in physical contact with these chemicals. For your safety, please read the enclosed Material Safety Data Sheets.

When conducting soil sampling, please clean off any residual soil from the outside of the containers. This will help prevent cross contamination of other samples in the cooler.

Please fill out all sample identification tags as completely as possible.

Please fill out the enclosed Chain of Custody form for adequate sample tracking.

The temperature requirement for the receipt of most environmental samples is 4 ±2° C. Temperatures that exceed this range are subject to qualification and data rejection by regulatory agencies. Following the instructions below provides the best chance of achieving and maintaining this temperature and avoiding qualified data.

- Samples should be collected and placed on ice as soon as possible. It is much more difficult to cool down warm samples.
- When possible, sample containers should be sealed in zip-lock containers. This prevents cross contamination and protects the sample labels from moisture that could render them illegible.
- Do not overfill the cooler with samples. Overfilling the cooler limits the space available for ice.
- Surround the sides and the tops of the sample containers with loose, cubed, ice. Surrounding the samples with ice is the most efficient way of cooling them. Do not use individual small bags of ice. Do not simply lay a bag of ice on top of the samples.
- Place the temperature blank in a representative location in the cooler, not in the middle of a bag of ice.
- Secure all paperwork in a zip-lock bag and place in the cooler. Seal the cooler closed.
- When shipping the coolers back to TriMatrix, complete the enclosed FedEx Airbill and attach it to the cooler. Samples shipped during the week for standard overnight delivery typically arrive the next day between 9:00 and 10:00 a.m. Saturday deliveries must be approved by your project chemist. When shipping samples for a Saturday delivery, select Priority Overnight and Saturday Delivery on the FedEx Airbill.

Please call your TriMatrix project chemist at 1-616-975-4500 if you require any further instructions, or to notify them of the pending arrival of any non-scheduled samples.

Thank You,
TriMatrix Laboratories, Inc.

bp sample cooler packing.doc | Page 1 of 1 | revision: 0.0

Attachment 20.13
Example - Commercial Invoice for International Shipments

TriMatrix Laboratories, Inc.

Commercial Invoice

International
Air Waybill No. Master #

Date of Exportation 12/10/08	**Export References:** Gary Wood **Caribe GE Vieques**
Shipper/Exporter John Shipper TriMatrix Laboratories Inc. 5560 Corporate Exchange Ct. Grand Rapids, MI 49512 (616) 975-4500	**Consignee: Nestor Rios** ABC Manufacturing PR 200 & PR 201 BO. Martineau Vieques, Puerto Rico 00765 (787) 741-7216
Country of Export U.S.A.	**Importer-If other than Consignee** **(Complete name and address)**
Country of Manufacture U.S.A.	
Country of Ultimate Destination Puerto Rico	

No. of Pkgs.	Type of Pkg.	Full description of Goods	Qty.	Unit Weight	Total Weight	Unit Value	Total Value
3 Coolers	Plastic	coolers	3	8.50lbs	25.50 lbs	$50.0	$150.00
	Glass	Sample container	141	0.10 lbs	14.10 lbs	$1.50	$211.50
	plastic	Sample container	17	0.10 lbs	1.70 lbs	$1.50	$25.50
	glass	Sample container filled w/ Deionized water	10	1.00lbs	10.00 lbs	$1.50	$15.00
	plastic	Sample container filled w/ tap water	2	1.50lbs	2.00 lbs	$1.50	$3.00
	plastic	Ziplock filled w/ paperwork	1	1.50 lbs	1.50 lbs	$1.50	$1.50
Total Pkgs. 3					**Total Weight 54.80 lbs**		**Total Invoice $406.50 USA Value**

These Commodities, Technology, or software exported from the United States in accordance with the export administration Regulations. Diversion contrary to U.S. Law Prohibited.
I declare all the information contained in this invoice to be true and correct.

Signature of Shipper/Exporter *Date*

Attachment 20.14
Example – Instruction Sheet for USDA Regulated Soil Materials

TriMatrix Laboratories, Inc.

INSTRUCTIONS FOR THE HANDLING AND SHIPMENT OF USDA REGULATED SOIL MATERIALS

It is the responsibility the party shipping any USDA regulated soil material to TriMatrix Laboratories, Inc. in Grand Rapids, Michigan; to ship in accordance with DOT regulations if shipped within the U.S. and/ or the International Air Transportation Association (I.A.T.A), if shipped from outside the U.S.

Included in this shipment are three copies of the TriMatrix soil import permit (S-41980), and two copies of the USDA PPQ Form 550.

The following steps must be employed prior to shipment of regulated soil material to TriMatrix:

1. Include three copies of the TriMatrix Laboratory soil import permit with each cooler containing the regulated soil material. One copy must be given to the courier, the second copy is placed with paperwork inside of the cooler and the third and final copy is placed in a secured envelope outside the cooler labeled "USDA paperwork enclosed".

2. Include two copies of PPQ Form 550 with each cooler shipped with USDA regulated material enclosed. The first copy will be placed in the envelope labeled "USDA paperwork enclosed", and the second copy will be taped in a visible area on the outside of the cooler.

3. The words "Contents-Soil Samples" MUST be written in a visible area on the outside of cooler.

Any samples shipped to TriMatrix from a USDA regulated area that do not conform to these guidelines may be rejected.

Please call 1-616-975-4500 and speak to your project chemist with any questions.

USDA Soil Shipping Instructions | Page 1 of 1 | Revision 1.0

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Laboratory Waste Disposal

APPROVALS:

Area Supervisor: _____ Date: 5/22/06
Gary L. Wood

QA Officer: _____ Date: 5-22-06
Tom C. Boocher

Operations Manager: _____ Date: 5/22/06
Jeff P. Glaser

Procedure Number: GR-15-102
Revision Number: 2.1

Date Initiated: 9/30/03
Effective Date: 6/10/06

Date Revised: 5/18/06
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 18

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4-30-09		4-30-10

1.0 SCOPE AND APPLICATION

1.1 Correct disposal of laboratory waste is important from environmental and regulatory standpoints.

1.2 This procedure specifies general criteria used in laboratory waste characterization and disposal.

2.0 PRINCIPLE METHOD REFERENCES

2.1 Hazardous Waste Management Program Administrative Rules Promulgated Pursuant To Part 111, Hazardous Waste Management of Michigan's Natural Resources and Environmental Protection Act, 1994 PA 451, as amended (ACT 451)

2.2 Code of Federal Regulations, Title 40, *Environmental Protection Agency*, Chapter 1, Part 261.20 – 261.24, Identification and Listing of Hazardous Waste

2.3 Code of Federal Regulations, Title 49, *Environmental Protection Agency*, Chapter 1, Part 173.201 – 173.205, Shipping Papers

2.4 Code of Federal Regulations, Title 40, *Environmental Protection Agency*, Chapter 1, Part 261.31, Hazardous Wastes from Non-Specific Sources

3.0 SUMMARY OF PROCEDURE

3.1 Laboratory waste is generated from unused client samples, analysis by-products, used acids, used solvents, used oils and expired chemicals.

3.2 Each waste type requires a specific disposal. Accurate laboratory waste characterization is essential for proper disposal and to prevent safety and/or environmental hazards.

3.3 Most of the laboratory waste is segregated, composited, stored, packed and disposed of by an approved and certified waste disposal company.

3.4 All hazardous waste is manifested by the disposal company, and is required by federal and state regulations.

4.0 PARAMETER OR COMPOUND LIST

4.1 There are no parameters or compounds lists directly associated with this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-01-119, *Toxicity Characteristic Leaching Procedure (TCLP)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 There are no interferences or corrective procedures directly associated with this procedure.

7.0 SAFETY PRECAUTIONS

7.1 Caution must be used at all times when handling laboratory waste.

7.1.1 Most laboratory waste contains hazardous materials.

7.1.2 Most client samples may be corrosive, flammable, reactive or toxic from characteristic materials and/or from preservation chemicals.

7.1.3 Approved personal protective equipment (PPE) must be worn when handling any laboratory waste.

7.2 Personal protective equipment includes approved safety glasses, a laboratory coat and disposable gloves.

7.3 Packaging of individual waste containers into lab-packs for transport and disposal is performed only by the disposal company.

7.4 All TriMatrix employees must read and understand the TriMatrix Safety Manual and Chemical Hygiene Plan. A copy of these documents is given to each new employee at the safety orientation.

7.5 All TriMatrix employees have access to the Material Safety Data Sheet (MSDS) library located on the laboratory intranet.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Client sample disposal is to be done only after the scheduled disposal time in Element unless otherwise specified.

8.2 With pre-approval, extremely hazardous, highly concentrated and/or toxic samples must be returned to the client unless otherwise specified.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Laboratory Information Management System, Promium-ELEMENT™

9.2 Jars, "TCLP", 4-liter glass or plastic

9.3 ELEMENT "Samples Eligible For Disposal" report

9.4 Boxes and/or overpack drums, D.O.T. approved, drums are supplied by the waste hauler

9.5 Waste manifests, MDEQ, supplied by the waste hauler

9.6 Hazardous waste storage and accumulation labels

9.7 Hach EZ-COD Recycling Vessel, 20 gallon, part 2895420

9.8 pH paper, wide-range

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no routine preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Concentrated solutions of acid and sodium hydroxide for pH treatment.

12.0 STANDARDS PREPARATION

12.1 There is no standards preparation directly associated with this procedure.

13.0 ANALYTICAL PROCEDURE

13.1 Analyzed client samples that are ready for disposal are the largest source of laboratory waste. Identify, characterize and dispose of sample waste as follows:

13.1.1 Using ELEMENT™, print a list of all samples eligible for disposal (Attachment 1).

13.1.1.1 At the main menu select "Sample Control", "Disposal" and "Print Samples Eligible for Disposal Report". Refer to Attachment 1 for a report example.

13.1.1.2 Refer to the flow chart provided in Attachment 2.

13.1.2 The list printed in Section 13.1.1 only identifies samples logged into ELEMENT™. Samples not logged in such as those put on hold by a client will not be listed. Check the "hold shelves" but consult the project chemist for disposal instructions of any sample "on hold".

13.1.3 Water or wastewater sample waste can be disposed of by pouring down the drain after pH adjustment unless otherwise specified. Soil, oil, sludge and waste samples must follow strict disposal requirements and cannot be disposed of to the city sewer.

13.1.4 The ELEMENT™ disposal report flags all samples if a contaminant exceeds a regulatory disposal level. A default disposal level of 10,000 ppm will also trigger a flag for non-regulated

contaminants. No flagged sample is to be disposed of through the municipal sewer or landfill (drain or garbage bin).

13.1.5 The federal Environmental Protection Agency (EPA) has classified hazardous waste based on characteristic definitions and a list of toxic contaminants considered to be hazardous to the health and/or environment when present above certain concentrations. TriMatrix Laboratories, as a small quantity waste generator, is required to dispose of any laboratory waste as hazardous which has the characteristic definition of ignitability (D001), corrosivity (D002), or reactivity (D003). The laboratory is also required to dispose of laboratory waste as hazardous if compounds present on the TCLP toxicity characteristic list are present in the waste above the regulatory level. All hazardous waste disposals must be manifested. Disposal of D001-D003 hazardous waste is summarized below. Refer to Attachment 3, the Toxicity Characteristic List, to determine the characteristic of toxicity. A waste is considered hazardous whether it exhibits one characteristic or all. Any sample designated as hazardous is disposed of through the hazardous waste company or is returned to the client. The preference is to return any hazardous sample to the client. However, the project chemist must determine whether this action is appropriate. If a hazardous sample can not be returned to the client, it must be classified as waste and disposed of through the hazardous waste company. When determining whether sample waste is hazardous, check for ignitability, corrosivity and reactivity first since these wastes are specific classifications and are quickly determined. Then, determine characteristic toxicity.

13.1.5.1 Ignitability (D001): A sample must be returned to the client or manifested for disposal as ignitable hazardous waste if it meets any of the following criteria:

13.1.5.1.1 If it is a liquid, other than an aqueous solution containing less than 24 percent alcohol by volume and has a flashpoint less than 60 °C (140 °F).

13.1.5.1.2 It is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture or spontaneous chemical changes and when ignited, burns so vigorously and persistently that it creates a hazard.

13.1.5.1.3 It is an ignitable compressed gas.

13.1.5.1.4 It is an ozidizer.

13.1.5.2 Corrosivity (D002): A sample must be returned to the client or manifested for disposal as corrosive hazardous waste if it meets any of the following criteria:

13.1.5.2.1 It is aqueous and has a pH that is less than 2.

13.1.5.2.2 It is aqueous and has a pH that is greater than 12.5.

13.1.5.2.3 It is a liquid and corrodes steel (SAE 1020) at a rate of more than 6.35 mm (0.250 in) per year at a test temperature of 55° C (130° F).

NOTE: The exception to the above is that wastewaters and many other aqueous samples can be treated to neutralize the pH. Disposal can then be through the municipal sewer if no other hazardous classification is found or unless otherwise specified.

13.1.5.3 Reactivity (D003): A sample must be returned to the client or manifested for disposal as reactive hazardous waste if it meets any of the following criteria:

13.1.5.3.1 It is normally unstable and readily undergoes violent change without detonating.

13.1.5.3.2 It reacts violently with water.

13.1.5.3.3 It forms potentially explosive mixtures with water.

13.1.5.3.4 When mixed with water, it generates toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

13.1.5.3.5 It is a cyanide or sulfide-bearing waste that, when exposed to pH conditions between 2.0 and 12.5, can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

13.1.5.3.6 It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.

13.1.5.3.7 It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.

13.1.5.3.8 It is a forbidden explosive or it meets the definition of a class 1/division 1.1, 1.2 or 1.3 explosive.

13.1.5.4 Toxicity Characteristics (D004 – D043):

13.1.5.4.1 A sample must be returned to the client or manifested for disposal as toxic hazardous waste if it exhibits the corresponding "D" characteristic of toxicity. The characteristic of toxicity is determined based on the TCLP extract from a representative aliquot containing any TCLP contaminant equal to or greater than the regulatory limit given in Attachment 3. Refer to TriMatrix SOP GR-01-119 for details of the TCLP extraction.

13.1.5.4.1.1 Do not put soil samples confirmed to be D004-D043 hazardous waste into the non-hazardous soils composite drum or the municipal dumpster. The

hazardous classification does not go away by mixing with non-hazardous soils and the drum or dumpster will automatically take on the hazardous classification by association.

13.1.5.4.1.2 If a "totals" analysis is run on a soil sample and the result divided by 20 is less than the TCLP regulatory limit, the sample may be classified as not toxic for the TCLP parameter analyzed.

13.1.5.4.1.3 If a "totals" analysis is run on a soil sample and the result divided by 20 is greater than the regulatory level, the sample remains unclassified for toxicity for the parameter analyzed, but may be presumed as suspect for disposal purposes.

13.1.5.4.1.4 If a "totals" analysis is run on a liquid sample, the result may be presumed to be the TCLP result and compared to the TCLP regulatory limit.

13.1.5.3 Listed Waste (F, K, P, U): A sample must be returned to the client or manifested for disposal as listed hazardous waste if it meets any of the following criteria:

13.1.5.3.1 It is received by the laboratory as a hazardous waste sample from a non-specific source (F-waste).

13.1.5.3.2 It is received by the laboratory as a hazardous waste sample from a specific source (K-waste).

13.1.5.3.3 It is received by the laboratory as a hazardous waste sample from a discarded commercial chemical product or species(P-waste and U-waste).

13.1.5.4 Polychlorinated Biphenyls Waste (PCB): A sample must be returned to the client or manifested for disposal as PCB hazardous waste if it meets any of the following criteria:

13.1.5.4.1 It is received by the laboratory as a hazardous waste sample containing PCBs at any concentration.

13.1.5.4.2 It is analyzed and found to contain PCBs at any concentration.

13.1.5.4.3 Soil or oil waste composites found to contain PCBs at any concentration.

13.1.6 Samples considered non-hazardous may be disposed of by way of the city sewer or municipal dumpster if the following criteria are met:

13.1.6.1 The sample is not ignitable.

13.1.6.2 The sample is not corrosive.

13.1.6.3 The sample is not reactive.

13.1.6.4 The sample has been tested and is not toxic for all TCLP parameters or is verified to be non-toxic for all TCLP parameters through client, project chemist and/or analyst experience.

13.1.6.5 The sample was not received from the client as a listed hazardous waste.

13.1.7 Samples considered hazardous may be returned to the client as follows:

13.1.7.1 Return all samples in the original tagged sample container. Do not remove the sample tag.

13.1.7.2 Wrap sample containers in bubble wrap to prevent breakage, leaking, spilling or vaporization then package in a DOT-approved box or cooler. D.O.T approved containers are specified in 49CFR Ch. 1, Part 173.201-173.205 and generally identified as such on the container.

13.1.7.3 Assure that the following information accompanies the sample if it is not on the sample container label and/or tag:

13.1.7.2.1 The client's name, mailing address, and telephone number

13.1.7.2.2 The laboratory's name, mailing address, and telephone number

13.1.7.2.3 The approximate quantity of the sample.

13.1.7.2.4 The date of shipment.

13.1.7.2.5 A description of the sample.

13.1.7.4 Ship the package in compliance with DOT regulations as a laboratory sample.

13.1.8 Samples not returned to the client and considered or suspected as being hazardous must be disposed of through the hazardous waste disposal company as follows:

13.1.8.1 If a sample is confirmed to be hazardous by any TCLP parameter, D001-D003 characteristic, process listing or contains PCBs, store together with other "like" samples in TCLP jars in the waste storage room until a waste haul is scheduled. Label each TCLP jar with the appropriate internal hazardous waste storage label (Refer to Attachment 4).

13.1.8.2 Non-ignitable, non-corrosive and non-reactive soil sample waste that is presumed to be non-toxic based on the sample source and/or prior client/project chemist/analyst experience is to be compiled in a labeled drum and stored in the flammables cabinet area of the garage. However, before placing in the drum, each sample must be individually tested and found non-hazardous

for D001-D003 characteristics. Forward a copy of the results to the safety officer if not a part of the client-requested analysis. As the drum fills, homogenize the contents by mixing. When full, take a representative sampling and schedule for a full TCLP analysis. If TCLP results on the sampling indicate the contents are non-toxic, the drum may be disposed of through the hazardous waste disposal company as non-notification, non-hazardous material. Forward a copy of the TCLP results to the safety officer to file with the manifest.

13.1.8.3 Non-aqueous liquid sample waste will generally be classified as hazardous based on the client's testing request or sample identification. However, oils and solvents not testing as being hazardous must be tentatively classified based on client information and/or analyst experience before placing in the waste storage room. Oil samples must be checked for polychlorinated biphenyls (PCB) or confirmed by the client to not have PCBs. Forward a copy of the PCB analysis (if not an analysis requested by the client) to the safety officer. Label each sample appropriately for disposal by the hazardous waste disposal company.

13.1.8.4 Aqueous liquid sample waste will generally be classified as wastewater when not found to have a hazardous constituent and can be disposed of by way of the municipal sewer after neutralizing the pH.

13.2 Analysis By-Product Waste

13.2.1 Each analysis by-product waste requires individualized classification for disposal by the hazardous waste disposal company.

13.2.1.1 Chemical Oxygen Demand (COD) By-Products

13.2.1.1.1 COD by-product material contains sulfuric acid and compounds of silver, chromium, and mercury which can be recovered. As vials become available from the analysis, they are to be deposited in the Hach EZ-COD™ recycling vessel. The 20-gallon drum is located in the chemical storage area at the rear of the garage. Full containers of spent COD vials are then picked-up by Heritage Environmental for recycling. This material is not manifested as waste when sent to recycling.

13.2.1.2 Cyanide Distillation Waste

13.2.1.2.1 Cyanide distillation waste is comprised of sulfuric acid. The waste is collected in a 4-liter bottle labeled as sulfuric acid waste. A start date is put on the bottle. When the bottle becomes full, write the filled date on the label and store until the next waste haul. This waste is manifested as Waste Corrosive Liquid, Toxic, NOS, 8(6.1), UN2922, PG II (Sulfuric Acid).

13.2.1.3 Flash Point Waste

13.2.1.3.1 Liquid and solid flash point waste is tested remnants that flash at less than 140° F. Composite and store these remnants in the waste cabinet, in a glass TCLP jar with a PTFE-lined lid. Composite PCB-containing sample remnants separately.

13.2.1.3.2 Disposal will generally be manifested similar to "Waste Solid Containing Flammable Liquid, NOS, 4.1, UN3175, Pg II, (Methanol, Soil)".

13.2.1.4 pH Wastes

13.2.1.4.1 Complile aqueous remnants of pH testing as wastewater and dispose of according to Section 13.1.8.4.

13.2.1.4.2 Compile solid remnants of pH testing as soil and dispose of according to Section 13.1.8.2.

13.2.1.5 Solvent Wastes

13.2.1.5.1 Solvent wastes are generated in the individual lab areas through various processes. Solvents fall into two main categories, halogenated and non-halogenated and are composited as such. Identify the solvent contained in each bottle on the internal hazardous waste storage label. For example, a halogenated solvent bottle containing methylene chloride must be labeled "Halogenated – Methylene Chloride". A non-halogenated bottle containing hexane/toluene must be labeled "Non-Halogenated – Hexane/Toluene". Also label each bottle with a start and end date. The waste is manifested as "Waste Flammable Liquid, Toxic, NOS, 3(6.1), UN1992, PGII, (Acetone, Methylene Chloride)" and "Waste Flammable Liquid, NOS, UN1993, PGII, (Acetone, Toluene, Hexane)".

13.2.1.5.2 Methanol waste from soil extractions in the Volatile Organics laboratory are composited and stored in satellite jars. All containers are labeled as "MeOH waste" and must have an accumulation start and end date.

13.2.1.5.3 Solvent waste containing PCBs (except as specified in Section 13.2.1.7) or chloroform must be composited and stored in separate bottles for disposal. Do not mix PCB waste and chloroform however. Label each bottle with an internal hazardous waste storage label as PCB or Chloroform waste, including the start and end date. Store filled bottles in the flammables waste cabinet. The PCB waste is manifested as "Polychlorinated biphenyls, Liquid, 9, UN2315, PGII, (PCB Samples)" and the chloroform waste as "Waste Chloroform, 6.1, UN1888, Pg II".

13.2.1.6 Acid Wastes

13.2.1.6.1 Different types of acids are used in the laboratory. Collect each type in separate waste bottles and label appropriately (chromerge, sulfuric, or nitric). Also record the start and end date on each bottle. Full bottles must be disposed of through the hazardous waste disposal company. This waste is manifested as "Waste Corrosive Liquid, Toxic, NOS, 8(6.1), UN2922, PGII, (Constituent Acid, Chromic Acid).

13.2.1.7 PCB Wastes

13.2.1.7.1 Any material containing PCBs must be considered hazardous and disposed of as such. Label each bottle as PCB waste, including a start and end date. PCB wastes must only be composited with other PCB waste. ANY material with PCBs must be segregated from other waste and compiled based on compatibility and matrix. The waste is generally manifested as "Polychlorinated Biphenyls, Solid, NOS, 9, UN2315, PGII, (PCB: SOILS)".

13.2.1.8 Semi-Volatile Extracts

13.2.1.8.1 Extract vials are concentrated contaminants from samples analyzed in the extraction and semi-volatile laboratories. Vials are lab-packed and disposed of by the hazardous waste disposal company. However, vials containing any PCB concentration must be stored with the PCB waste (Section 13.2.1.7) for disposal as PCB waste. Non-PCB vials are manifested as "Waste Flammable Liquids, Toxic, NOS, 3, UN1992, PGII, (Methylene Chloride, Hexane)".

13.2.1.9 Outdated Chemicals and Standards

13.2.1.9.1 Outdated chemicals must remain in the original container and be lab-packed by the hazardous waste disposal company for disposal. When removed from the laboratory, store in the flammables cabinet for outdated chemicals until disposal. Manifesting will be at the discretion of the disposal company.

13.2.1.9.2 As an alternative, outdated chemicals may be donated to a credible institution or industrial laboratory without manifesting.

13.2.1.10 Chemical Spills

13.2.1.10.1 Chemical spills must be packed individually and labeled in accordance with the material and hazard class collected.

13.2.1.10.2 Manifesting will be at the discretion of the disposal company.

13.3 As the allotted waste storage space fills, arrangements for a waste haul must be made. TriMatrix uses a certified waste disposal company for all hazardous waste disposals. All hazardous waste manifests are completed by the disposal company as the waste is transferred to the disposal company's truck. Manifest copies are sent to the state of Michigan as required then maintained on file by the safety officer.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Manifesting is described in Section 13.0 and an example is presented as Attachment 5.

14.2 All analyses for the purpose of waste characterization and/or disposal must be copied to the laboratory safety officer for maintaining on file.

15.0 QUALITY ASSURANCE

15.1 There is no quality assurance directly associated with this procedure.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 There is no demonstration of capability study and/or method validation directly associated with this procedure.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

18.3 Follow all instructions in this procedure for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 Hazardous Waste Management Program Administrative Rules Promulgated Pursuant To Part 111, Hazardous Waste Management of Michigan's Natural Resources and Environmental Protection Act, 1994 PA 451, as amended (ACT 451)

19.2 Code of Federal Regulations, Title 40, *Environmental Protection Agency*, Chapter 1, Part 261.20 – 261.24, Identification and Listing of Hazardous Waste

19.3 Code of Federal Regulations, Title 49, *Environmental Protection Agency*, Chapter 1, Part 173.201 – 173.205, Shipping Papers

19.4 Code of Federal Regulations, Title 40, *Environmental Protection Agency*, Chapter 1, Part 261.31, Hazardous Wastes from Non-Specific Sources

20.0 ATTACHMENTS

20.1 Attachment 1: Samples Eligible for Disposal Report Example

20.2 Attachment 2: Sample Disposal Flowchart

20.3 Attachment 3: Toxicity Characteristic List

20.4 Attachment 4: Internal Hazardous Waste Storage label Example

20.7 Attachment 5: MDEQ Waste Manifest Example

ATTACHMENT 1
Samples Eligible for Disposal Report Example

TriMatrix Laboratories, Inc.

DISPOSAL REPORT -- Page 1 of 4

Printed 5/22/2006, 11:49:20AM

Samples Ready for disposal, received between Apr-24-06 and May-08-06

All clients; All projects; All matrices; All containers; All locations

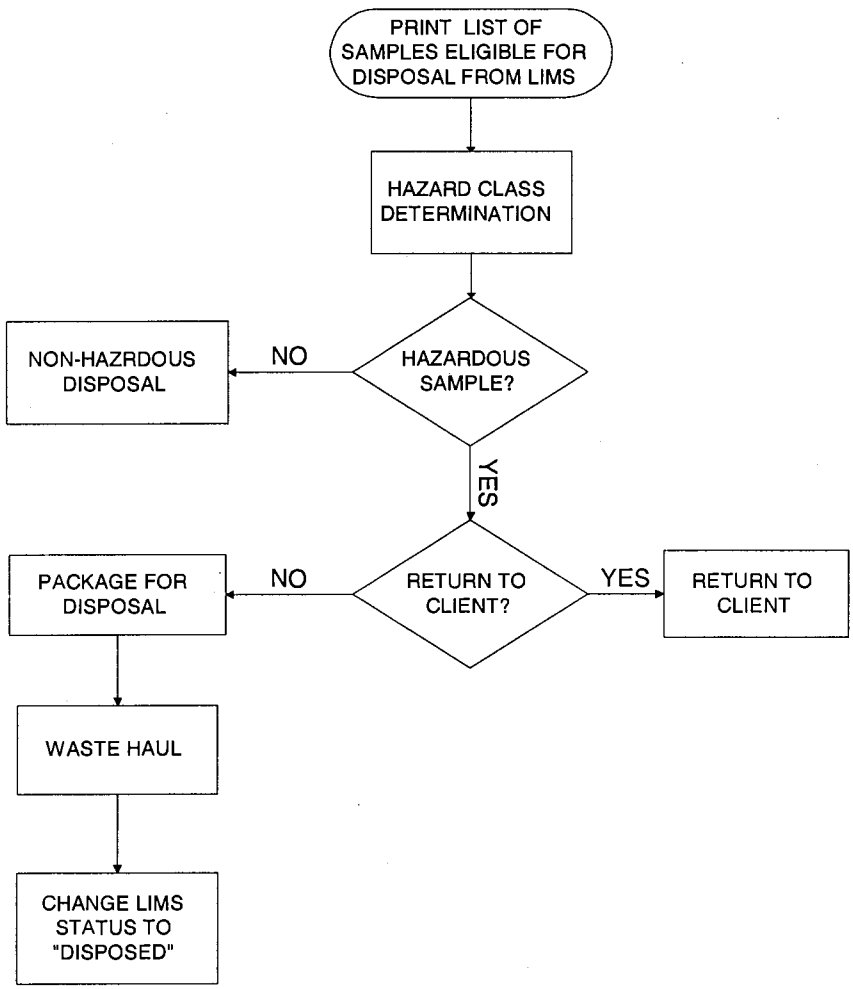
Work Order	Sample	Matrix	Client / Hazard	Project	LabMgr	Status	Status Date
0604418			Taste Control Consultants	Waste Analysis	LMH	Completed	May-02-06 14:05
0604419			Smith & Jones Engineering	Beauford Oil - Emmett Street	LMH	Completed	May-08-06 17:44
0604420			Detroit Machinery - Plant-2	Chem Pond - Monitoring Point 009A	LMH	Completed	May-03-06 12:39
0604423	03	Waste	HJ - Metal Center **Cadmium TCLP [1.6mg/L]**	Waste Characterization	JLR	Completed	May-05-06 14:34
0604433			Smith & Jones Engineering	Fred Oil - Emmett Street	LMH	Completed	May-08-06 17:46
0604434			Smith & Jones Engineering	Yankee Springs	LMH	Completed	May-02-06 18:28
0604435			HJ - Engine Plant	Program 9 - Daily PCB Monitoring	JLR	Completed	Apr-27-06 10:37
0604437			Global Research & Development	PT-02 Quarterly Monitoring	JLR	Completed	May-01-06 10:53
0604438			Jones Associates, Inc.	Total Coliform	GLW	Completed	May-03-06 15:27
0604440			Detroit Machinery - RRPP	River Water Analysis	LMH	Completed	May-02-06 18:48
0604443			AAA Corporation	Wastewater Monitoring, Kentwood Facility	WJR	Completed	May-01-06 12:07
0604446			Hill Valley BPU	Wastewater Monitoring Program	GLW	Completed	May-04-06 14:48
0604447			Worthington Orchard Co.	Weekly Wastewater	LMH	Completed	May-04-06 19:49
0604448			GGH, Inc - Dublin, OH	New Waste Characterization	JLR	Completed	May-09-06 10:26
0604449			Oak Tree Semiconductor Corp.	Monthly Industrial Discharge	JLR	Completed	May-03-06 12:02
0604450			Jones Associates, Inc.	Total Coliform	GLW	Completed	May-03-06 15:27
0604451			Thomas County DPW	First Twp. - Phosphorus Study	JLR	Completed	May-01-06 10:52
0604452			Thomas County DPW	First Township; Permit #MI0036547	JLR	Completed	May-08-06 08:58
0604453			Edison Transmission Plant	Wastewater Monitoring	TLM	Completed	May-03-06 15:36
0604455			Alaska Proving Ground	Weekly 001 Monitoring	TLM	Completed	May-03-06 14:18
0604456			Sherman Clocks	Wastewater Monitoring	JLR	Completed	May-03-06 11:59

dsp_TriMatrix.rpt

Approved By: 5-22-06 **QA Officer**

Approved By: 5/22/06 **Area Supervisor**

ATTACHMENT 2
Sample Disposal Flowchart



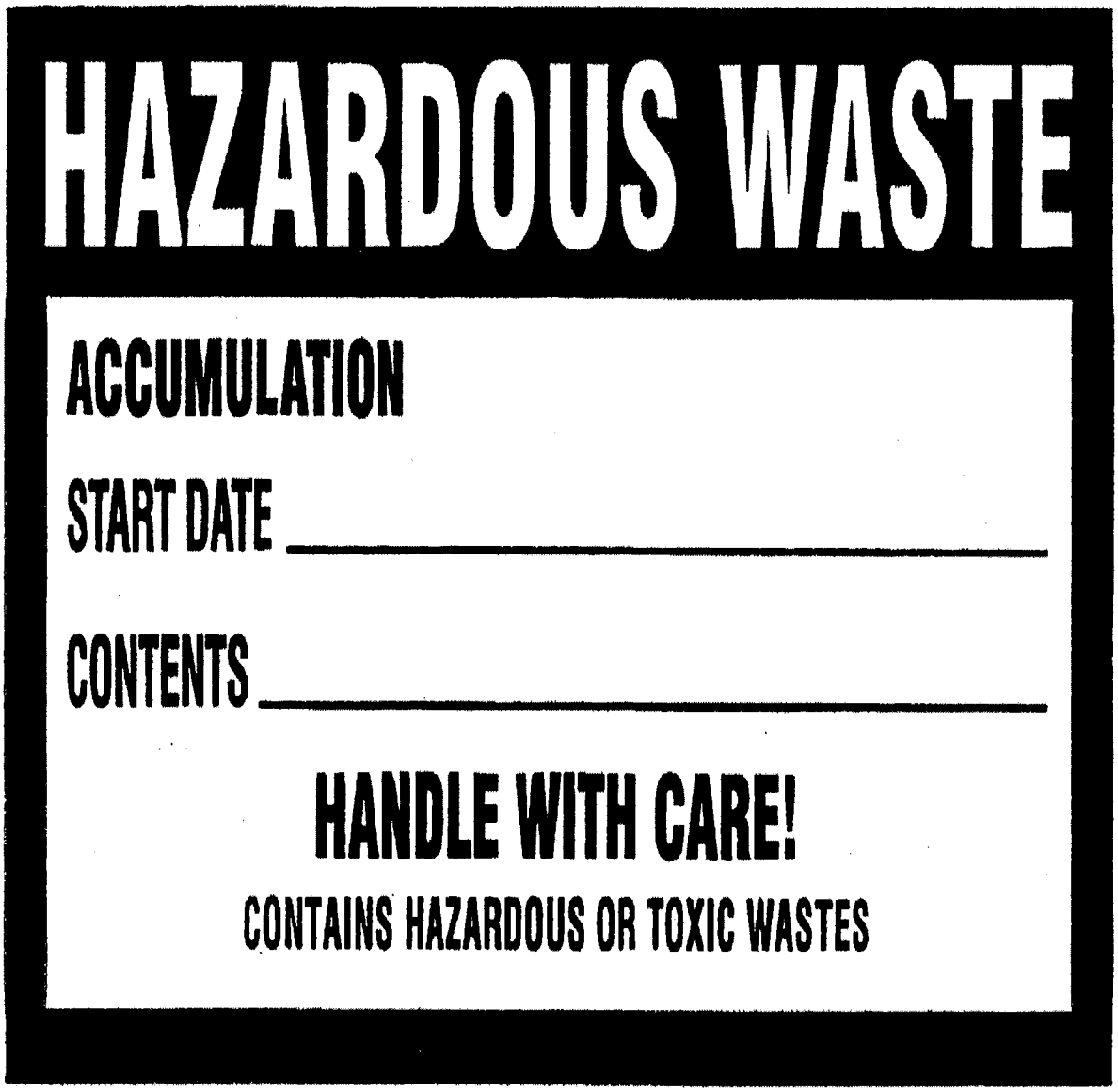
Approved By: 5-22-06 **QA Officer** **Approved By:** 5/22/06 **Area Supervisor**

ATTACHMENT 3
Toxicity Characteristic List

EPA Hazardous Waste #	Contaminant	Total Regulatory Level (mg/kg)	TCLP Level (mg/L)
D004	Arsenic	100	5.0
D005	Barium	2000	100.0
D018	Benzene	10	0.5
D006	Cadmium	20	1.0
D019	Carbon Tetrachloride	10	0.5
D020	Chlordane	.60	0.03
D021	Chlorobenzene	2000	100.0
D022	Chloroform	120	6.0
D007	Chromium	100	5.0
D023	o-Cresol	4000	200.0
D024	m-Cresol	4000	200.0
D025	p-Cresol	4000	200.0
D026	Cresol	4000	200.0
D016	2,4,-D	200	10.0
D027	1,4-Dichlorobenzene	150	7.5
D028	1,2-Dichloroethane	10	0.5
D029	1,1-Dichloroethylene	14	0.7
D030	2,4-Dinitrotoluene	2.60	0.13
D012	Endrin	.40	0.02
D031	Heptachlor	.160	0.008
D032	Hexachlorobenzene	22.6	1.13
D033	Hexachlorobutadiene	10.0	0.5
D034	Hexachloroethane	60.0	3.0
D008	Lead	100.0	5.0
D013	Lindane	8.0	0.4
D009	Mercury	4.0	0.2
D014	Methoxychlor	200.00	10.0
D035	Methyl Ethyl Ketone	4000.00	200.0
D036	Nitrobenzene	40.0	2.0
D037	Pentachlorophenol	2000.0	100.0
D038	Pyridine	100.0	5.0
D010	Selenium	20.0	1.0
D011	Silver	100.0	5.0
D039	Tetrachloroethylene	14.0	0.7
D015	Toxaphene	10.0	0.5
D040	Trichloroethylene	10.0	0.5
D041	2,4,5-Trichlorophenol	8000	400.0
D042	2,4,6-Trichlorophenol	40.0	2.0
D017	2,4,5-TP (Silvex)	20.0	1.0
D043	Vinyl Chloride	4.0	0.2
	Cyanide	320	16
	PCBs (polychlorinated biphenyls)	<50 mg/kg from the source	

Approved By: _____ 5-22-06 _____ Approved By: _____ 5/22/06 _____
QA Officer Area Supervisor

ATTACHMENT 4
Internal Hazardous Waste Storage Label Example



Approved By: _____ 5-22-06 Approved By: _____ 5/22/06
QA Officer Area Supervisor

ATTACHMENT 5
MDEQ Waste Manifest Example

DEQ WASTE MANAGEMENT DIVISION
MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

DO NOT WRITE IN THIS SPACE
ATT. ☐ DIS. ☐ REJ. ☐ PR. ☐

Required under authority of Part 111 and Part 121 of Act 451, 1994, as amended.

Failure to file may subject you to criminal and/or civil penalties under Sections 324.11151 or 324.12116 MCL.

Please print or type. Form Approved. OMB No. 2050-0039

UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator's US EPA ID No.: MIR 000 041 632
Manifest Document No.: 02640
2. Page 1 of 1
Information in the shaded areas is not required by Federal law.

3. Generator's Name and Mailing Address: TriMatrix Laboratories, Inc.
5560 Corporate Exchange Court
Grand Rapids, MI 49512
4. Generator's Phone ((616))975-4500

A. State Manifest Document Number: MI 10102640
B. State Generator's ID:
C. State Transporter's ID:
D. Transporter's Phone: (269) 685-9824
E. State Transporter's ID:
F. Transporter's Phone:
G. State Facility's ID:
H. Facility's Phone: (269) 685-9824

5. Transporter 1 Company Name: Drug & Laboratory Disposal, Inc.
6. US EPA ID Number: MID092947928
7. Transporter 2 Company Name:
8. US EPA ID Number:
9. Designated Facility Name and Site Address: Drug & Laboratory Disposal, Inc.
331 Broad Street
Plainwell, MI 49080-1439
10. US EPA ID Number: MID092947928

	HM	11. US DOT Description (including Proper Shipping Name, Hazard Class, and ID NUMBER).	12. Containers No.	Type	13. Total Quantity	14. Unit Wt/Vol	I. Waste No.
a.	X	WASTE FLAMMABLE LIQUID, n.o.s., 3, UN1993, PG II (ACETONITRILE, METHANOL)	1	DF	80	P	F003
b.	X	WASTE FLAMMABLE LIQUID, TOXIC, n.o.s., 3(6.1), UN1992, PG II (ACETONE, METHYLENE CHLORIDE) RQ=D001	3	DF	320	P	F003
c.	X	WASTE CHLOROFORM, 6.1, UN1888, PG III RQ=10#	1	DF	50	P	D022
d.	X	WASTE SOLID CONTAINING FLAMMABLE LIQUID, n.o.s., 4.1, UN3175, PG II (METHANOL, SOIL) RQ=D001	1	DF	130	P	F003

J. Additional Descriptions for Materials Listed Above
a) LP, D001
b) LP, F002, D001, D022
c) LP
d) LP, D001

K. Handling Codes
a
b
c
d

15. Special Handling Instructions and Additional Information
In case of accident, contain material and call (269) 685-9825. ERG guidebook on vehicle. DLD has the appropriate permits for, and will accept, the above wastes.

16. GENERATOR'S CERTIFICATION: I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national government regulations. If I am a large quantity generator, I certify that I have a program in place to reduce the volume and toxicity of waste generated to the degree I have determined to be economically practicable and that I have selected the practicable method of treatment storage, or disposal currently available to me which minimizes the present and future threat to human health and the environment; OR; if I am a small quantity generator, I have made a good faith effort to minimize my waste generation and select the best waste management method that is available to me and that I can afford.

Printed/Typed Name: Joshua Frank
Signature: Joshua Frank
Date: Month 01 Day 20 Year 06

17. Transporter 1 Acknowledgment of Receipt of Materials
Printed/Typed Name: KYLE GOODWIN
Signature: Kyle Goodwin
Date: Month 01 Day 20 Year 06

18. Transporter 2 Acknowledgment of Receipt of Materials
Printed/Typed Name:
Signature:
Date: Month Day Year

19. Discrepancy Indication Space

20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.
Printed/Typed Name: Kevin Jay Berghuis, CHMM
Signature:
Date: Month Day Year

GENERATOR / TRANSPORTER / FACILITY

ALL SPILLS MUST BE REPORTED TO THE MICHIGAN POLLUTION EMERGENCY ALERTING SYSTEM, IN MICHIGAN AT 1-800-292-4706 OR OUT OF STATE AT 517-373-7660 AND THE NATIONAL RESPONSE CENTER AT 1-800-424-8802 24 HOUR PER DAY.

EPA Form 8700-22 (Rev. 9/88)

To be mailed by Generator to: WASTE MANAGEMENT DIVISION, MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY, PO BOX 30038, LANSING MI 48909-7538

EQP 5110 Rev. 7/03

Approved By: [signature] 5-22-06 QA Officer
Approved By: [signature] 5/22/06 Area Supervisor

Table 28.1 Quality Control Limits - TriMatrix Laboratories, Inc.

Method	Analyte	Surr. %R	DUP RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike %R	Blank Spike RPD	CAS #
Extractable Petroleum Hydrocarbons by EPA Method 8015B in Soil								
USEPA-8015B	ERO - 8015B (C9-C36)	-	20	50-150	30	50-150	30	
USEPA-8015B	o-Terphenyl	44-137	-	-	-	-	-	84-15-1
Extractable Petroleum Hydrocarbons by EPA Method 8015B in Water								
USEPA-8015B	ERO - 8015B (C8-C36)	-	20	50-150	20	50-150	20	
USEPA-8015B	o-Terphenyl	45-136	-	-	-	-	-	84-15-1
Volatile Petroleum Hydrocarbons by EPA Method 8015B in Soil								
USEPA-8015B	GRO - 8015B (C5-C12)	-	30	72-118	20	72-118	30	
USEPA-8015B	aaa-Trifluorotoluene	76-113	-	-	-	-	-	98-08-8
Volatile Petroleum Hydrocarbons by EPA Method 8015B in Water								
USEPA-8015B	GRO - 8015B (C5-C12)	-	20	75-122	20	75-122	20	
USEPA-8015B	aaa-Trifluorotoluene	86-113	-	-	-	-	-	98-08-8
Polychlorinated Biphenyls (PCBs) by EPA Method 8082 in Soil								
USEPA-8082	PCB-1016	-	-	40-140	30	40-140	30	12674-11-2
USEPA-8082	PCB-1221	-	-	-	-	-	-	11104-28-2
USEPA-8082	PCB-1232	-	-	-	-	-	-	11141-16-5
USEPA-8082	PCB-1242	-	-	-	-	-	-	53469-21-9
USEPA-8082	PCB-1248	-	-	-	-	-	-	12672-29-6
USEPA-8082	PCB-1254	-	-	-	-	-	-	11097-69-1
USEPA-8082	PCB-1260	-	-	60-130	30	60-130	30	11096-82-5
USEPA-8082	PCB-1262	-	-	-	-	-	-	37324-23-5
USEPA-8082	PCB-1268	-	-	-	-	-	-	11100-14-4
USEPA-8082	Decachlorobiphenyl	60-125	-	-	-	-	-	2051-24-3
USEPA-8082	Tetrachloro-m-xylene	32-129	-	-	-	-	-	877-09-8
Polychlorinated Biphenyls (PCBs) by EPA Method 8082 in Water								
USEPA-8082	PCB-1016	-	-	25-145	30	25-145	30	12674-11-2
USEPA-8082	PCB-1221	-	-	-	-	-	-	11104-28-2
USEPA-8082	PCB-1232	-	-	-	-	-	-	11141-16-5
USEPA-8082	PCB-1242	-	-	-	-	-	-	53469-21-9
USEPA-8082	PCB-1248	-	-	-	-	-	-	12672-29-6
USEPA-8082	PCB-1254	-	-	-	-	-	-	11097-69-1
USEPA-8082	PCB-1260	-	-	30-145	30	30-145	30	11096-82-5
USEPA-8082	PCB-1262	-	-	-	-	-	-	37324-23-5
USEPA-8082	PCB-1268	-	-	-	-	-	-	11100-14-4
USEPA-8082	Decachlorobiphenyl	40-135	-	-	-	-	-	2051-24-3
USEPA-8082	Tetrachloro-m-xylene	36-114	-	-	-	-	-	877-09-8
Organochlorine Pesticides by EPA Method 8081A in Soil								
USEPA-8081A	alpha-BHC	-	47	60-125	30	60-125	30	319-84-6
USEPA-8081A	beta-BHC	-	51	60-125	30	60-125	30	319-85-7
USEPA-8081A	gamma-BHC (Lindane)	-	45	60-125	30	60-125	30	58-89-9

Table 28.1 Quality Control Limits - TriMatrix Laboratories, Inc.

Method	Analyte	Surr. %R	DUP RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike %R	Blank Spike RPD	CAS #
USEPA-8081A	delta-BHC	-	54	55-130	30	55-130	30	319-86-8
USEPA-8081A	alpha-Chlordane	-	20	65-120	30	65-120	30	5103-71-9
USEPA-8081A	gamma-Chlordane	-	20	65-125	30	65-125	30	5103-74-2
USEPA-8081A	4,4'-DDD	-	20	30-135	30	30-135	30	72-54-8
USEPA-8081A	4,4'-DDE	-	20	70-125	30	70-125	30	72-55-9
USEPA-8081A	4,4'-DDT	-	47	45-140	30	45-140	30	50-29-3
USEPA-8081A	Aldrin	-	58	45-140	30	45-140	30	309-00-2
USEPA-8081A	Dieldrin	-	46	65-125	30	65-125	30	60-57-1
USEPA-8081A	Endosulfan I	-	20	15-135	30	15-135	30	959-98-8
USEPA-8081A	Endosulfan II	-	20	35-140	30	35-140	30	33213-65-9
USEPA-8081A	Endosulfan Sulfate	-	73	60-135	30	60-135	30	1031-07-8
USEPA-8081A	Endrin	-	52	60-135	30	60-135	30	72-20-8
USEPA-8081A	Endrin Aldehyde	-	74	35-145	30	35-145	30	7421-93-4
USEPA-8081A	Endrin Ketone	-	20	65-135	30	65-135	30	53494-70-5
USEPA-8081A	Heptachlor	-	48	50-140	30	50-140	30	76-44-8
USEPA-8081A	Heptachlor Epoxide	-	35	65-130	30	65-130	30	1024-57-3
USEPA-8081A	Methoxychlor	-	20	55-145	30	55-145	30	72-43-5
USEPA-8081A	Toxaphene	-	20	40-150	30	40-150	30	8001-35-2
USEPA-8081A	Tetrachloro-m-xylene	70-125	-	-	-	-	-	877-09-8
USEPA-8081A	Decachlorobiphenyl	55-130	-	-	-	-	-	2051-24-3
Organochlorine Pesticides by EPA Method 8081A in Water								
USEPA-8081A	4,4'-DDD	-	20	25-150	30	25-150	30	72-54-8
USEPA-8081A	4,4'-DDE	-	20	35-140	30	35-140	30	72-55-9
USEPA-8081A	4,4'-DDT	-	20	45-140	30	45-140	30	50-29-3
USEPA-8081A	Aldrin	-	20	25-140	30	25-140	30	309-00-2
USEPA-8081A	alpha-BHC	-	20	60-130	30	60-130	30	319-84-6
USEPA-8081A	alpha-Chlordane	-	20	65-125	30	65-125	30	5103-71-9
USEPA-8081A	beta-BHC	-	20	65-125	30	65-125	30	319-85-7
USEPA-8081A	delta-BHC	-	20	45-135	30	45-135	30	319-86-8
USEPA-8081A	Dieldrin	-	20	60-130	30	60-130	30	60-57-1
USEPA-8081A	Endosulfan I	-	20	50-110	30	50-110	30	959-98-8
USEPA-8081A	Endosulfan II	-	20	30-130	30	30-130	30	33213-65-9
USEPA-8081A	Endosulfan Sulfate	-	20	55-135	30	55-135	30	1031-07-8
USEPA-8081A	Endrin	-	20	55-135	30	55-135	30	72-20-8
USEPA-8081A	Endrin Aldehyde	-	20	55-135	30	55-135	30	7421-93-4
USEPA-8081A	Endrin Ketone	-	20	75-125	30	75-125	30	53494-70-5
USEPA-8081A	gamma-BHC (Lindane)	-	20	25-135	30	25-135	30	58-89-9
USEPA-8081A	gamma-Chlordane	-	20	60-125	30	60-125	30	5103-74-2
USEPA-8081A	Heptachlor	-	20	40-130	30	40-130	30	76-44-8
USEPA-8081A	Heptachlor Epoxide	-	20	60-130	30	60-130	30	1024-57-3
USEPA-8081A	Methoxychlor	-	20	55-150	30	55-150	30	72-43-5
USEPA-8081A	Toxaphene	-	20	40-145	30	40-145	30	8001-35-2
USEPA-8081A	Tetrachloro-m-xylene	25-140	-	-	-	-	-	877-09-8
USEPA-8081A	Decachlorobiphenyl	30-135	-	-	-	-	-	2051-24-3
Volatile Organic Compounds by EPA Method 8260B in Soil								
USEPA-8260B	1,1,1-Trichloroethane	-	30	70-135	30	70-135	30	71-55-6
USEPA-8260B	1,1,2,2-Tetrachloroethane	-	30	55-130	30	55-130	30	79-34-5
USEPA-8260B	1,1,2-Trichloroethane	-	30	60-125	30	60-125	30	79-00-5
USEPA-8260B	1,1-Dichloroethane	-	30	75-125	30	75-125	30	75-34-3
USEPA-8260B	1,1-Dichloroethene	-	30	65-135	30	65-135	30	75-35-4

Table 28.1 Quality Control Limits - TriMatrix Laboratories, Inc.

Method	Analyte	Surr. %R	DUP RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike %R	Blank Spike RPD	CAS #
USEPA-8260B	1,2,4-Trichlorobenzene	-	30	65-130	30	65-130	30	120-82-1
USEPA-8260B	1,2-Dibromo-3-chloropropan	-	30	40-135	30	40-135	30	96-12-8
USEPA-8260B	1,2-Dibromoethane	-	30	70-125	30	70-125	30	106-93-4
USEPA-8260B	1,2-Dichlorobenzene	-	30	75-120	30	75-120	30	95-50-1
USEPA-8260B	1,2-Dichloroethane	-	30	70-135	30	70-135	30	107-06-2
USEPA-8260B	1,2-Dichloropropane	-	30	70-120	30	70-120	30	78-87-5
USEPA-8260B	1,3-Dichlorobenzene	-	30	70-125	30	70-125	30	541-73-1
USEPA-8260B	1,4-Dichlorobenzene	-	30	70-125	30	70-125	30	106-46-7
USEPA-8260B	2-Butanone (MEK)	-	30	30-160	30	30-160	30	78-93-3
USEPA-8260B	2-Hexanone	-	30	45-145	30	45-145	30	591-78-6
USEPA-8260B	4-Methyl-2-pentanone (MIBK	-	30	45-145	30	45-145	30	108-10-1
USEPA-8260B	Acetone	-	30	20-160	30	20-160	30	67-64-1
USEPA-8260B	Benzene	-	30	75-125	30	75-125	30	71-43-2
USEPA-8260B	Bromodichloromethane	-	30	70-130	30	70-130	30	75-27-4
USEPA-8260B	Bromoform	-	30	55-135	30	55-135	30	75-25-2
USEPA-8260B	Bromomethane	-	30	30-160	30	30-160	30	74-83-9
USEPA-8260B	Carbon Disulfide	-	30	45-160	30	45-160	30	75-15-0
USEPA-8260B	Carbon Tetrachloride	-	30	65-135	30	65-135	30	56-23-5
USEPA-8260B	Chlorobenzene	-	30	75-125	30	75-125	30	108-90-7
USEPA-8260B	Chloroethane	-	30	40-155	30	40-155	30	75-00-3
USEPA-8260B	Chloroform	-	30	70-125	30	70-125	30	67-66-3
USEPA-8260B	Chloromethane	-	30	50-130	30	50-130	30	74-87-3
USEPA-8260B	cis-1,2-Dichloroethene	-	30	65-125	30	65-125	30	156-59-2
USEPA-8260B	cis-1,3-Dichloropropene	-	30	70-125	30	70-125	30	10061-01-5
USEPA-8260B	Cyclohexane	-	30	70-130	30	70-130	30	110-82-7
USEPA-8260B	Dibromochloromethane	-	30	65-130	30	65-130	30	124-48-1
USEPA-8260B	Dichlorodifluoromethane	-	30	35-135	30	35-135	30	75-71-8
USEPA-8260B	Ethylbenzene	-	30	75-125	30	75-125	30	100-41-4
USEPA-8260B	Isopropylbenzene	-	30	75-130	30	75-130	30	98-82-8
USEPA-8260B	Methyl Acetate	-	30	70-130	30	70-130	30	79-20-9
USEPA-8260B	Methyl tert-Butyl Ether	-	30	63-127	30	63-127	30	1634-04-4
USEPA-8260B	Methylcyclohexane	-	30	70-130	30	70-130	30	108-87-2
USEPA-8260B	Methylene Chloride	-	30	55-140	30	55-140	30	75-09-2
USEPA-8260B	Styrene	-	30	75-125	30	75-125	30	100-42-5
USEPA-8260B	Tetrachloroethene	-	30	65-140	30	65-140	30	127-18-4
USEPA-8260B	Toluene	-	30	70-125	30	70-125	30	108-88-3
USEPA-8260B	trans-1,2-Dichloroethene	-	30	65-135	30	65-135	30	156-60-5
USEPA-8260B	trans-1,3-Dichloropropene	-	30	65-125	30	65-125	30	10061-02-6
USEPA-8260B	Trichloroethene	-	30	75-125	30	75-125	30	79-01-6
USEPA-8260B	Trichlorofluoromethane	-	30	25-185	30	25-185	30	75-69-4
USEPA-8260B	Vinyl Chloride	-	30	60-125	30	60-125	30	75-01-4
USEPA-8260B	Xylene (Total)	-	30	75-125	30	75-125	30	1330-20-7
USEPA-8260B	Dibromofluoromethane	78-121	-	-	-	-	-	1868-53-7
USEPA-8260B	1,2-Dichloroethane-d4	66-124	-	-	-	-	-	17060-07-0
USEPA-8260B	Toluene-d8	85-115	-	-	-	-	-	2037-26-5
USEPA-8260B	4-Bromofluorobenzene	85-120	-	-	-	-	-	460-00-4
USEPA-8260B	Fluorobenzene	-	-	-	-	-	-	462-06-6
USEPA-8260B	Chlorobenzene-d5	-	-	-	-	-	-	3114-55-4
USEPA-8260B	1,4-Dichlorobenzene-d4	-	-	-	-	-	-	3855-82-1
Volatile Organic Compounds by EPA Method 8260B in Water								
USEPA-8260B	1,1,1-Trichloroethane	-	30	65-130	30	65-130	30	71-55-6
USEPA-8260B	1,1,2,2-Tetrachloroethane	-	30	65-130	30	65-130	30	79-34-5

Table 28.1 Quality Control Limits - TriMatrix Laboratories, Inc.

		Surr.	DUP	Matrix Spike		Blank Spike		
Method	**Analyte**	**%R**	**RPD**	**%R**	**RPD**	**%R**	**RPD**	**CAS #**
USEPA-8260B	1,1,2-Trichloroethane	-	30	75-125	30	75-125	30	79-00-5
USEPA-8260B	1,1-Dichloroethane	-	30	70-135	30	70-135	30	75-34-3
USEPA-8260B	1,1-Dichloroethene	-	30	70-130	30	70-130	30	75-35-4
USEPA-8260B	1,2,4-Trichlorobenzene	-	30	65-135	30	65-135	30	120-82-1
USEPA-8260B	1,2-Dibromo-3-chloropropan	-	30	50-130	30	50-130	30	96-12-8
USEPA-8260B	1,2-Dibromoethane	-	30	80-120	30	80-120	30	106-93-4
USEPA-8260B	1,2-Dichlorobenzene	-	30	70-120	30	70-120	30	95-50-1
USEPA-8260B	1,2-Dichloroethane	-	30	70-130	30	70-130	30	107-06-2
USEPA-8260B	1,2-Dichloropropane	-	30	75-125	30	75-125	30	78-87-5
USEPA-8260B	1,3-Dichlorobenzene	-	30	75-125	30	75-125	30	541-73-1
USEPA-8260B	1,4-Dichlorobenzene	-	30	75-125	30	75-125	30	106-46-7
USEPA-8260B	2-Butanone (MEK)	-	30	30-150	30	30-150	30	78-93-3
USEPA-8260B	2-Hexanone	-	30	55-130	30	55-130	30	591-78-6
USEPA-8260B	4-Methyl-2-pentanone (MIBK	-	30	60-135	30	60-135	30	108-10-1
USEPA-8260B	Acetone	-	30	40-140	30	40-140	30	67-64-1
USEPA-8260B	Benzene	-	30	80-120	30	80-120	30	71-43-2
USEPA-8260B	Bromodichloromethane	-	30	75-120	30	75-120	30	75-27-4
USEPA-8260B	Bromoform	-	30	70-130	30	70-130	30	75-25-2
USEPA-8260B	Bromomethane	-	30	30-145	30	30-145	30	74-83-9
USEPA-8260B	Carbon Disulfide	-	30	35-160	30	35-160	30	75-15-0
USEPA-8260B	Carbon Tetrachloride	-	30	65-140	30	65-140	30	56-23-5
USEPA-8260B	Chlorobenzene	-	30	80-120	30	80-120	30	108-90-7
USEPA-8260B	Chloroethane	-	30	60-135	30	60-135	30	75-00-3
USEPA-8260B	Chloroform	-	30	65-135	30	65-135	30	67-66-3
USEPA-8260B	Chloromethane	-	30	40-125	30	40-125	30	74-87-3
USEPA-8260B	cis-1,2-Dichloroethene	-	30	70-125	30	70-125	30	156-59-2
USEPA-8260B	cis-1,3-Dichloropropene	-	30	70-130	30	70-130	30	10061-01-5
USEPA-8260B	Cyclohexane	-	30	77-125	30	77-125	30	110-82-7
USEPA-8260B	Dibromochloromethane	-	30	60-135	30	60-135	30	124-48-1
USEPA-8260B	Dichlorodifluoromethane	-	30	30-155	30	30-155	30	75-71-8
USEPA-8260B	Ethylbenzene	-	30	75-125	30	75-125	30	100-41-4
USEPA-8260B	Isopropylbenzene	-	30	75-125	30	75-125	30	98-82-8
USEPA-8260B	Methyl Acetate	-	30	70-130	30	70-130	30	79-20-9
USEPA-8260B	Methyl tert-Butyl Ether	-	30	65-125	30	65-125	30	1634-04-4
USEPA-8260B	Methylcyclohexane	-	30	70-130	30	70-130	30	108-87-2
USEPA-8260B	Methylene Chloride	-	30	55-140	30	55-140	30	75-09-2
USEPA-8260B	Styrene	-	30	65-135	30	65-135	30	100-42-5
USEPA-8260B	Tetrachloroethene	-	30	45-150	30	45-150	30	127-18-4
USEPA-8260B	Toluene	-	30	75-120	30	75-120	30	108-88-3
USEPA-8260B	trans-1,2-Dichloroethene	-	30	60-140	30	60-140	30	156-60-5
USEPA-8260B	trans-1,3-Dichloropropene	-	30	55-140	30	55-140	30	10061-02-6
USEPA-8260B	Trichloroethene	-	30	70-125	30	70-125	30	79-01-6
USEPA-8260B	Trichlorofluoromethane	-	30	60-145	30	60-145	30	75-69-4
USEPA-8260B	Vinyl Chloride	-	30	50-145	30	50-145	30	75-01-4
USEPA-8260B	Xylene (Total)	-	30	75-130	30	75-130	30	1330-20-7
USEPA-8260B	Dibromofluoromethane	85-115	30	-	-	-	-	1868-53-7
USEPA-8260B	1,2-Dichloroethane-d4	70-120	30	-	-	-	-	17060-07-0
USEPA-8260B	Toluene-d8	85-120	30	-	-	-	-	2037-26-5
USEPA-8260B	4-Bromofluorobenzene	75-120	30	-	-	-	-	460-00-4
USEPA-8260B	Fluorobenzene	-	-	-	-	-	-	462-06-6
USEPA-8260B	Chlorobenzene-d5	-	-	-	-	-	-	3114-55-4
USEPA-8260B	1,4-Dichlorobenzene-d4	-	-	-	-	-	-	3855-82-1

Semivolatile Organic Compounds by EPA Method 8270C

Table 28.1 Quality Control Limits - TriMatrix Laboratories, Inc.

Method	Analyte	Surr. %R	DUP RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike %R	Blank Spike RPD	CAS #
in Soil								
USEPA-8270C	1,1'-Biphenyl	-	30	60-131	30	60-131	30	92-52-4
USEPA-8270C	2,4,5-Trichlorophenol	-	30	50-110	30	50-110	30	95-95-4
USEPA-8270C	2,4,6-Trichlorophenol	-	30	45-110	30	45-110	30	88-06-2
USEPA-8270C	2,4-Dichlorophenol	-	30	45-110	30	45-110	30	120-83-2
USEPA-8270C	2,4-Dimethylphenol	-	30	30-105	30	30-105	30	105-67-9
USEPA-8270C	2,4-Dinitrophenol	-	30	15-130	30	15-130	30	51-28-5
USEPA-8270C	2,4-Dinitrotoluene	-	30	50-115	30	50-115	30	121-14-2
USEPA-8270C	2,6-Dinitrotoluene	-	30	50-110	30	50-110	30	606-20-2
USEPA-8270C	2-Chloronaphthalene	-	30	45-105	30	45-105	30	91-58-7
USEPA-8270C	2-Chlorophenol	-	30	45-105	30	45-105	30	95-57-8
USEPA-8270C	2-Methylnaphthalene	-	30	45-105	30	45-105	30	91-57-6
USEPA-8270C	2-Methylphenol	-	30	40-105	30	40-105	30	95-48-7
USEPA-8270C	2-Nitroaniline	-	30	45-120	30	45-120	30	88-74-4
USEPA-8270C	2-Nitrophenol	-	30	40-110	30	40-110	30	88-75-5
USEPA-8270C	3,3´-Dichlorobenzidine	-	30	10-130	30	10-130	30	91-94-1
USEPA-8270C	3-Nitroaniline	-	30	25-110	30	25-110	30	99-09-2
USEPA-8270C	4,6-Dinitro-2-methylphenol	-	30	30-135	30	30-135	30	534-52-1
USEPA-8270C	4-Bromophenyl Phenyl Ether	-	30	45-115	30	45-115	30	101-55-3
USEPA-8270C	4-Chloro-3-methylphenol	-	30	45-115	30	45-115	30	59-50-7
USEPA-8270C	4-Chloroaniline	-	30	10-95	30	10-95	30	106-47-8
USEPA-8270C	4-Chlorophenyl Phenyl Ether	-	30	45-110	30	45-110	30	7005-72-3
USEPA-8270C	4-Methylphenol	-	30	40-105	30	40-105	30	106-44-5
USEPA-8270C	4-Nitroaniline	-	30	35-115	30	35-115	30	100-01-6
USEPA-8270C	4-Nitrophenol	-	30	15-140	30	15-140	30	100-02-7
USEPA-8270C	Acetophenone	-	30	50-150	30	50-150	30	98-86-2
USEPA-8270C	Atrazine	-	30	61-146	30	61-146	30	1912-24-9
USEPA-8270C	Benzaldehyde	-	30	50-150	30	50-150	30	100-52-7
USEPA-8270C	Benzidine	-	30	10-74	30	10-74	30	92-87-5
USEPA-8270C	Bis(2-chloroethoxy)methane	-	30	45-110	30	45-110	30	111-91-1
USEPA-8270C	Bis(2-chloroethyl) Ether	-	30	40-105	30	40-105	30	111-44-4
USEPA-8270C	Bis(2-chloroisopropyl) Ether	-	30	20-115	30	20-115	30	108-60-1
USEPA-8270C	Bis(2-ethylhexyl) Phthalate	-	30	45-125	30	45-125	30	117-81-7
USEPA-8270C	Butyl Benzyl Phthalate	-	30	50-125	30	50-125	30	85-68-7
USEPA-8270C	Caprolactam	-	30	62-112	30	62-112	30	105-60-2
USEPA-8270C	Carbazole	-	30	45-115	30	45-115	30	86-74-8
USEPA-8270C	Dibenzofuran	-	30	50-105	30	50-105	30	132-64-9
USEPA-8270C	Diethyl Phthalate	-	30	50-115	30	50-115	30	84-66-2
USEPA-8270C	Dimethyl Phthalate	-	30	50-110	30	50-110	30	131-11-3
USEPA-8270C	Di-n-butyl Phthalate	-	30	55-110	30	55-110	30	84-74-2
USEPA-8270C	Di-n-octyl Phthalate	-	30	40-130	30	40-130	30	117-84-0
USEPA-8270C	Hexachlorobenzene	-	30	45-120	30	45-120	30	118-74-1
USEPA-8270C	Hexachlorobutadiene	-	30	40-115	30	40-115	30	87-68-3
USEPA-8270C	Hexachlorocyclopentadiene	-	30	10-113	30	10-113	30	77-47-4
USEPA-8270C	Hexachloroethane	-	30	35-110	30	35-110	30	67-72-1
USEPA-8270C	Indeno(1,2,3-cd)pyrene	-	30	40-120	30	40-120	30	193-39-5
USEPA-8270C	Isophorone	-	30	45-110	30	45-110	30	78-59-1
USEPA-8270C	Nitrobenzene	-	30	40-115	30	40-115	30	98-95-3
USEPA-8270C	N-Nitroso-di-n-propylamine	-	30	40-115	30	40-115	30	621-64-7
USEPA-8270C	N-Nitroso-diphenylamine	-	30	50-115	30	50-115	30	86-30-6
USEPA-8270C	Pentachlorophenol	-	30	25-120	30	25-120	30	87-86-5
USEPA-8270C	Phenol	-	30	40-100	30	40-100	30	108-95-2
USEPA-8270C	2-Fluorophenol	35-105	-	-	-	-	-	367-12-4
USEPA-8270C	Phenol-d6	40-100	-	-	-	-	-	13127-88-3

Table 28.1 Quality Control Limits - TriMatrix Laboratories, Inc.

Method	Analyte	Surr. %R	DUP RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike %R	Blank Spike RPD	CAS #
USEPA-8270C	Nitrobenzene-d5	35-100	-	-	-	-	-	4165-60-0
USEPA-8270C	2-Fluorobiphenyl	45-105	-	-	-	-	-	321-60-8
USEPA-8270C	2,4,6-Tribromophenol	35-125	-	-	-	-	-	118-79-6
USEPA-8270C	o-Terphenyl	30-125	-	-	-	-	-	84-15-1
USEPA-8270C	1,4-Dichlorobenzene-d4	-	-	-	-	-	-	3855-82-1
USEPA-8270C	Naphthalene-d8	-	-	-	-	-	-	1146-65-2
USEPA-8270C	Acenaphthene-d10	-	-	-	-	-	-	15067-26-2
USEPA-8270C	Phenanthrene-d10	-	-	-	-	-	-	1517-22-2
USEPA-8270C	Chrysene-d12	-	-	-	-	-	-	1719-03-5
USEPA-8270C	Perylene-d12	-	-	-	-	-	-	1520-96-3
Semivolatile Organic Compounds by EPA Method 8270C in Water								
USEPA-8270C	1,1'-Biphenyl	-	30	59-114	30	59-114	30	92-52-4
USEPA-8270C	2,4,5-Trichlorophenol	-	30	50-110	30	50-110	30	95-95-4
USEPA-8270C	2,4,6-Trichlorophenol	-	30	50-115	30	50-115	30	88-06-2
USEPA-8270C	2,4-Dichlorophenol	-	30	50-105	30	50-105	30	120-83-2
USEPA-8270C	2,4-Dimethylphenol	-	30	30-110	30	30-110	30	105-67-9
USEPA-8270C	2,4-Dinitrophenol	-	30	15-140	30	15-140	30	51-28-5
USEPA-8270C	2,4-Dinitrotoluene	-	30	50-120	30	50-120	30	121-14-2
USEPA-8270C	2,6-Dinitrotoluene	-	30	50-115	30	50-115	30	606-20-2
USEPA-8270C	2-Chloronaphthalene	-	30	50-105	30	50-105	30	91-58-7
USEPA-8270C	2-Chlorophenol	-	30	35-105	30	35-105	30	95-57-8
USEPA-8270C	2-Methylnaphthalene	-	30	45-105	30	45-105	30	91-57-6
USEPA-8270C	2-Methylphenol	-	30	40-110	30	40-110	30	95-48-7
USEPA-8270C	2-Nitroaniline	-	30	50-115	30	50-115	30	88-74-4
USEPA-8270C	2-Nitrophenol	-	30	40-115	30	40-115	30	88-75-5
USEPA-8270C	3,3´-Dichlorobenzidine	-	30	20-110	30	20-110	30	91-94-1
USEPA-8270C	3-Nitroaniline	-	30	20-125	30	20-125	30	99-09-2
USEPA-8270C	4,6-Dinitro-2-methylphenol	-	30	40-130	30	40-130	30	534-52-1
USEPA-8270C	4-Bromophenyl Phenyl Ether	-	30	50-115	30	50-115	30	101-55-3
USEPA-8270C	4-Chloro-3-methylphenol	-	30	45-110	30	45-110	30	59-50-7
USEPA-8270C	4-Chloroaniline	-	30	15-110	30	15-110	30	106-47-8
USEPA-8270C	4-Chlorophenyl Phenyl Ether	-	30	50-110	30	50-110	30	7005-72-3
USEPA-8270C	4-Methylphenol	-	30	30-110	30	30-110	30	106-44-5
USEPA-8270C	4-Nitroaniline	-	30	35-120	30	35-120	30	100-01-6
USEPA-8270C	4-Nitrophenol	-	30	0-125	30	0-125	30	100-02-7
USEPA-8270C	Acetophenone	-	30	54-113	30	54-113	30	98-86-2
USEPA-8270C	Atrazine	-	30	61-139	30	61-139	30	1912-24-9
USEPA-8270C	Benzaldehyde	-	30	25-141	30	25-141	30	100-52-7
USEPA-8270C	Bis(2-chloroethoxy)methane	-	30	45-105	30	45-105	30	111-91-1
USEPA-8270C	Bis(2-chloroethyl) Ether	-	30	35-110	30	35-110	30	111-44-4
USEPA-8270C	Bis(2-chloroisopropyl) Ether	-	30	25-130	30	25-130	30	108-60-1
USEPA-8270C	Bis(2-ethylhexyl) Phthalate	-	30	40-125	30	40-125	30	117-81-7
USEPA-8270C	Butyl Benzyl Phthalate	-	30	45-115	30	45-115	30	85-68-7
USEPA-8270C	Caprolactam	-	30	25-135	30	25-135	30	105-60-2
USEPA-8270C	Carbazole	-	30	50-115	30	50-115	30	86-74-8
USEPA-8270C	Dibenzofuran	-	30	55-105	30	55-105	30	132-64-9
USEPA-8270C	Diethyl Phthalate	-	30	40-120	30	40-120	30	84-66-2
USEPA-8270C	Dimethyl Phthalate	-	30	25-125	30	25-125	30	131-11-3
USEPA-8270C	Di-n-butyl Phthalate	-	30	55-115	30	55-115	30	84-74-2
USEPA-8270C	Di-n-octyl Phthalate	-	30	35-135	30	35-135	30	117-84-0
USEPA-8270C	Hexachlorobenzene	-	30	50-110	30	50-110	30	118-74-1
USEPA-8270C	Hexachlorobutadiene	-	30	25-105	30	25-105	30	87-68-3

Table 28.1 Quality Control Limits - TriMatrix Laboratories, Inc.

Method	Analyte	Surr. %R	DUP RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike %R	Blank Spike RPD	CAS #
USEPA-8270C	Hexachlorocyclopentadiene	-	30	30-141	30	30-141	30	77-47-4
USEPA-8270C	Hexachloroethane	-	30	30-95	30	30-95	30	67-72-1
USEPA-8270C	Indeno(1,2,3-cd)pyrene	-	30	45-125	30	45-125	30	193-39-5
USEPA-8270C	Isophorone	-	30	50-110	30	50-110	30	78-59-1
USEPA-8270C	Nitrobenzene	-	30	45-110	30	45-110	30	98-95-3
USEPA-8270C	N-Nitroso-di-n-propylamine	-	30	35-130	30	35-130	30	621-64-7
USEPA-8270C	N-Nitroso-diphenylamine	-	30	50-110	30	50-110	30	86-30-6
USEPA-8270C	Pentachlorophenol	-	30	40-115	30	40-115	30	87-86-5
USEPA-8270C	Phenol	-	30	0-115	30	0-115	30	108-95-2
USEPA-8270C	2-Fluorophenol	20-110	-	-	-	-	-	367-12-4
USEPA-8270C	Phenol-d6	10-115	-	-	-	-	-	13127-88-3
USEPA-8270C	Nitrobenzene-d5	40-110	-	-	-	-	-	4165-60-0
USEPA-8270C	2-Fluorobiphenyl	50-110	-	-	-	-	-	321-60-8
USEPA-8270C	2,4,6-Tribromophenol	40-125	-	-	-	-	-	118-79-6
USEPA-8270C	o-Terphenyl	50-135	-	-	-	-	-	84-15-1
USEPA-8270C	1,4-Dichlorobenzene-d4	-	-	-	-	-	-	3855-82-1
USEPA-8270C	Naphthalene-d8	-	-	-	-	-	-	1146-65-2
USEPA-8270C	Acenaphthene-d10	-	-	-	-	-	-	15067-26-2
USEPA-8270C	Phenanthrene-d10	-	-	-	-	-	-	1517-22-2
USEPA-8270C	Chrysene-d12	-	-	-	-	-	-	1719-03-5
USEPA-8270C	Perylene-d12	-	-	-	-	-	-	1520-96-3
Semivolatile Organic Compounds by EPA Method 8270C - Selective Ion Monitoring in Soil								
USEPA-8270C (SIM	1,4-Dioxane	-	-	30-120	30	30-120	30	123-91-1
USEPA-8270C (SIM	Acenaphthene	-	-	47-115	30	47-115	30	83-32-9
USEPA-8270C (SIM	Acenaphthylene	-	-	44-112	30	44-112	30	208-96-8
USEPA-8270C (SIM	Anthracene	-	-	42-123	30	42-123	30	120-12-7
USEPA-8270C (SIM	Benzo(a)anthracene	-	-	45-131	30	45-131	30	56-55-3
USEPA-8270C (SIM	Benzo(a)pyrene	-	-	36-130	30	36-130	30	50-32-8
USEPA-8270C (SIM	Benzo(b)fluoranthene	-	-	53-117	30	53-117	30	205-99-2
USEPA-8270C (SIM	Benzo(g,h,i)perylene	-	-	36-126	30	36-126	30	191-24-2
USEPA-8270C (SIM	Benzo(k)fluoranthene	-	-	52-115	30	52-115	30	207-08-9
USEPA-8270C (SIM	Chrysene	-	-	53-129	30	53-129	30	218-01-9
USEPA-8270C (SIM	Dibenz(a,h)anthracene	-	-	39-123	30	39-123	30	53-70-3
USEPA-8270C (SIM	Fluoranthene	-	-	43-123	30	43-123	30	206-44-0
USEPA-8270C (SIM	Fluorene	-	-	43-116	30	43-116	30	86-73-7
USEPA-8270C (SIM	Indeno(1,2,3-cd)pyrene	-	-	37-123	30	37-123	30	193-39-5
USEPA-8270C (SIM	Naphthalene	-	-	57-131	30	57-131	30	91-20-3
USEPA-8270C (SIM	Phenanthrene	-	-	45-122	30	45-122	30	85-01-8
USEPA-8270C (SIM	Pyrene	-	-	44-123	30	44-123	30	129-00-0
USEPA-8270C (SIM	Nitrobenzene-d5	40-132	-	-	-	-	-	4165-60-0
USEPA-8270C (SIM	2-Fluorobiphenyl	50-118	-	-	-	-	-	321-60-8
USEPA-8270C (SIM	o-Terphenyl	41-125	-	-	-	-	-	84-15-1
USEPA-8270C (SIM	1,4-Dichlorobenzene-d4	-	-	-	-	-	-	3855-82-1
USEPA-8270C (SIM	Naphthalene-d8	-	-	-	-	-	-	1146-65-2
USEPA-8270C (SIM	Acenaphthene-d10	-	-	-	-	-	-	15067-26-2
USEPA-8270C (SIM	Phenanthrene-d10	-	-	-	-	-	-	1517-22-2
USEPA-8270C (SIM	Chrysene-d12	-	-	-	-	-	-	1719-03-5
USEPA-8270C (SIM	Perylene-d12	-	-	-	-	-	-	1520-96-3
Semivolatile Organic Compounds by EPA Method 8270C - Selective Ion Monitoring in Water								
USEPA-8270C (SIM	1,4-Dioxane	-	-	30-120	30	30-120	30	123-91-1

Table 28.1 Quality Control Limits - TriMatrix Laboratories, Inc.

Method	Analyte	Surr. %R	DUP RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike %R	Blank Spike RPD	CAS #
USEPA-8270C (SI	Acenaphthene	-	-	39-109	20	39-109	20	83-32-9
USEPA-8270C (SI	Acenaphthylene	-	-	45-118	20	45-118	20	208-96-8
USEPA-8270C (SI	Anthracene	-	-	42-133	20	42-133	20	120-12-7
USEPA-8270C (SI	Benzo(a)anthracene	-	-	45-132	20	45-132	20	56-55-3
USEPA-8270C (SI	Benzo(a)pyrene	-	-	43-123	20	43-123	20	50-32-8
USEPA-8270C (SI	Benzo(b)fluoranthene	-	-	39-145	20	39-145	20	205-99-2
USEPA-8270C (SI	Benzo(g,h,i)perylene	-	-	47-126	20	47-126	20	191-24-2
USEPA-8270C (SI	Benzo(k)fluoranthene	-	-	61-118	20	61-118	20	207-08-9
USEPA-8270C (SI	Chrysene	-	-	46-133	20	46-133	20	218-01-9
USEPA-8270C (SI	Dibenz(a,h)anthracene	-	-	46-126	20	46-126	20	53-70-3
USEPA-8270C (SI	Fluoranthene	-	-	49-124	20	49-124	20	206-44-0
USEPA-8270C (SI	Fluorene	-	-	40-133	20	40-133	20	86-73-7
USEPA-8270C (SI	Indeno(1,2,3-cd)pyrene	-	-	42-128	20	42-128	20	193-39-5
USEPA-8270C (SI	Naphthalene	-	-	49-116	20	49-116	20	91-20-3
USEPA-8270C (SI	Phenanthrene	-	-	45-132	20	45-132	20	85-01-8
USEPA-8270C (SI	Pyrene	-	-	46-114	20	46-114	20	129-00-0
USEPA-8270C (SI	Nitrobenzene-d5	31-123	-	-	-	-	-	4165-60-0
USEPA-8270C (SI	2-Fluorobiphenyl	25-113	-	-	-	-	-	321-60-8
USEPA-8270C (SI	o-Terphenyl	42-125	-	-	-	-	-	84-15-1
USEPA-8270C (SI	1,4-Dichlorobenzene-d4	-	-	-	-	-	-	3855-82-1
USEPA-8270C (SI	Naphthalene-d8	-	-	-	-	-	-	1146-65-2
USEPA-8270C (SI	Acenaphthene-d10	-	-	-	-	-	-	15067-26-2
USEPA-8270C (SI	Phenanthrene-d10	-	-	-	-	-	-	1517-22-2
USEPA-8270C (SI	Chrysene-d12	-	-	-	-	-	-	1719-03-5
USEPA-8270C (SI	Perylene-d12	-	-	-	-	-	-	1520-96-3
Total Metals by EPA 6000/7000 Series Methods in Soil								
USEPA-6010B	Aluminum	-	20	80-120	20	80-120	20	7429-90-5
USEPA-6020A	Antimony	-	20	80-120	20	80-120	20	7440-36-0
USEPA-6020A	Arsenic	-	20	80-120	20	80-120	20	7440-38-2
USEPA-6020A	Barium	-	20	80-120	20	80-120	20	7440-39-3
USEPA-6020A	Beryllium	-	20	80-120	20	80-120	20	7440-41-7
USEPA-6020A	Cadmium	-	20	80-120	20	80-120	20	7440-43-9
USEPA-6010B	Calcium	-	20	80-120	20	80-120	20	7440-70-2
USEPA-6020A	Chromium	-	20	80-120	20	80-120	20	7440-47-3
USEPA-6020A	Cobalt	-	20	80-120	20	80-120	20	7440-48-4
USEPA-6020A	Copper	-	20	80-120	20	80-120	20	7440-50-8
USEPA-6010B	Iron	-	20	80-120	20	80-120	20	7439-89-6
USEPA-6020A	Lead	-	20	80-120	20	80-120	20	7439-92-1
USEPA-6010B	Magnesium	-	20	80-120	20	80-120	20	7439-95-4
USEPA-6020A	Manganese	-	20	80-120	20	80-120	20	7439-96-5
USEPA-7471A	Mercury	-	20	80-120	20	80-120	20	7439-97-6
USEPA-6020A	Nickel	-	20	80-120	20	80-120	20	7440-02-0
USEPA-6010B	Potassium	-	20	80-120	20	80-120	20	7440-09-7
USEPA-6020A	Selenium	-	20	80-120	20	80-120	20	7782-49-2
USEPA-6020A	Silver	-	20	75-120	20	75-120	20	7440-22-4
USEPA-6010B	Sodium	-	20	80-120	20	80-120	20	7440-23-5
USEPA-6020A	Thallium	-	20	80-120	20	80-120	20	7440-28-0
USEPA-6020A	Vanadium	-	20	80-120	20	80-120	20	7440-62-2
USEPA-6020A	Zinc	-	20	80-120	20	80-120	20	7440-66-6
Total Metals by EPA 6000/7000 Series Methods in Water								

Table 28.1 Quality Control Limits - TriMatrix Laboratories, Inc.

Method	Analyte	Surr. %R	DUP RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike %R	Blank Spike RPD	CAS #
USEPA-6010B	Aluminum	-	20	80-120	20	80-120	20	7429-90-5
USEPA-6020A	Antimony	-	20	80-120	20	80-120	20	7440-36-0
USEPA-6020A	Arsenic	-	20	80-120	20	80-120	20	7440-38-2
USEPA-6020A	Barium	-	20	80-120	20	80-120	20	7440-39-3
USEPA-6020A	Beryllium	-	20	80-120	20	80-120	20	7440-41-7
USEPA-6020A	Cadmium	-	20	80-120	20	80-120	20	7440-43-9
USEPA-6010B	Calcium	-	20	80-120	20	80-120	20	7440-70-2
USEPA-6020A	Chromium	-	20	80-120	20	80-120	20	7440-47-3
USEPA-6020A	Cobalt	-	20	80-120	20	80-120	20	7440-48-4
USEPA-6020A	Copper	-	20	80-120	20	80-120	20	7440-50-8
USEPA-6010B	Iron	-	20	80-120	20	80-120	20	7439-89-6
USEPA-6020A	Lead	-	20	80-120	20	80-120	20	7439-92-1
USEPA-6010B	Magnesium	-	20	80-120	20	80-120	20	7439-95-4
USEPA-6020A	Manganese	-	20	80-120	20	80-120	20	7439-96-5
USEPA-7470A	Mercury	-	20	80-120	20	80-120	20	7439-97-6
USEPA-6020A	Nickel	-	20	80-120	20	80-120	20	7440-02-0
USEPA-6010B	Potassium	-	20	80-120	20	80-120	20	7440-09-7
USEPA-6020A	Selenium	-	20	80-120	20	80-120	20	7782-49-2
USEPA-6020A	Silver	-	20	80-120	20	80-120	20	7440-22-4
USEPA-6010B	Sodium	-	20	80-120	20	80-120	20	7440-23-5
USEPA-6020A	Thallium	-	20	80-120	20	80-120	20	7440-28-0
USEPA-6020A	Vanadium	-	20	80-120	20	80-120	20	7440-62-2
USEPA-6020A	Zinc	-	20	80-120	20	80-120	20	7440-66-6

APPENDIX F

PA INVESTIGATION ANALYTICAL DATA SUMMARY TABLES AND DATA VALIDATION MEMO

ANALYTICAL RESULTS - DET ED COMPOUNDS ONLY
GROUNDWATER FROM TEST PITS
CODDINGTON COVE RUBBLE FILL AREA
NEWPORT, RHODE ISLAND

Sample Number		CCRF-GW-TP01		CCRF-GW-TP02		CCRF-GW-TP03		CCRF-GW-TP04		CCRF-A-DP01		CCRF-GW-TP05		CCRF-GW-TP08	
Sample Location		TP01		TP02		TP03		TP04		TP04		TP05		TP08	
Date Sampled		5/18/2004		5/18/2004		5/19/2004		5/19/2004		5/19/2004		5/19/2004		5/20/2004	
QC Identifier	MCL	None		None		None		Field Dup. CCRF-GW-TP04		Field Dup. CCRF-GW-TP04		None		None	
Volatile Organic Analysis (UG/L)															
1,1,2-Trichloro-1,2,2-trifluoroethane		5	U	11		5	U	5	U	5	U	5	U	5	U
Tetrachloroethene	5	5	U	2	J	5	U	5	U	5	U	5	U	5	U
TAL Metal Analysis (UG/L)															
Aluminum 37000		26200		10800		15600		11000		13200		16000		41100	
Arsenic	10	40.9		15.3		15.0		17.3		18.9		36.4		36.9	
Barium	2000	102	J	58.6	J	49.1	J	60.2	J	64.5	J	85.8	J	183	J
Calcium		41300		40100		83000		67300		67100		35800		123000	
Chromium	100	30.3		13.6		13.5		0.40	U	0.40	U	5.9		34.8	
Cobalt 11		23.0		15.7		30.3		14.7		15.6		26.2		46.6	
Copper	1300	58.9	U	34.9	U	39.7	U	15.4	U	16.0	U	38.0	U	124	
Iron		54600		26700		33800		62300		64200		83100		82800	
Lead	15	23.4		13.8		76.0		21.6		26.7		38.9		291	
Magnesium		18800		14900		12300		14000		14300		13400		18100	
Manganese 880		948		446		2220		5680		5640		5140		3980	
Mercury	2	0.13	U	0.14	U	0.14	U	0.13	U	0.13	U	0.14	U	0.35	
Nickel 730		53.6	J	28.6	J	29.2	J	0.53	UJ	2.7	UJ	15.2	J	60.2	J
Potassium		2550		1950		4660		2380		2520		2110		5760	
Selenium	50	6.8		4.0	U	4.0	U	4.0	U	4.0	U	5.2		4.0	U
Sodium		90600		89500		17700		16800		17100		32600		15700	
Vanadium 180		33.9	J	14.7	UJ	21.0	J	19.7	J	22.6	J	26.6	J	71.0	J
Zinc 11,000		118		87.5		96.0		37.8		39.2		68.0		284	

Black Background - Criteria Exceeded; U - Not detected; UJ - Detection limit approximate; J - Quantitation approximate;
R - Rejected; NA - Not Analyzed; * - From dilution analysis

ANALYTICAL RESULTS - DETE CONTAMINANTS ONLY
SURFACE SOIL AND TEST PIT SOIL SAMPLES
CODDINGTON COVE RUBBLE FILL AREA
NEWPORT, RHODE ISLAND

0008 0001 0-10

Sample Number		CCRF-S-SS01-0001		CCRF-S-SS02-0001		CCRF-S-SS03-0001		CCRF-S-SS04-0001		CCRF-S-SS05-0001		CCRF-S-SS06-0001		CCRF-S-TP01-0010		CCRF-S-TP02-0010		CCRF-S-DP01		CCRF-S-TP03-0010		CCRF-S-TP04-0010	
Sample Location		SS01		SS02		SS03		SS04		SS05		SS06		TP01		TP02		TP03		TP03		TP04	
Date Sampled		7/28/2004		7/28/2004		7/28/2004		7/28/2004		7/28/2004		7/28/2004		5/18/2004		5/18/2004		5/19/2004		5/19/2004		5/19/2004	
Interval		0.0-1.0		0.0-1.0		0.0-1.0		0.0-1.0		0.0-1.0		0.0-1.0		0.0-8.0		0.0-1.0		0.0-0.0		0.0-10.0		0.0-10.0	
QC Identifier	Region 9 PRG - Residential Soil_2002	None		Field Dup. CCRF-S-SS02-0001		None		None		None		None		None		None		Field Dup. CCRF-S-TP03-0010		Field Dup. CCRF-S-TP03-0010		None	
Volatile Organic Analysis (UG/KG)																							
1,4-Dichlorobenzene	3400	40	UJ	23	UJ	15	UJ	11	UJ	8	UJ	4	J	6	U	11	U	7	U	9	U	8	U
Acetone	1600000	1100	JEB	1800	JEB	29	JEB	30	JEB	40	JEB	130	JEB	6	U	11	U	19	JEB	14	EB	35	EB
Benzene	600	19	J	23	U	15	U	11	UJ	8	U	16	U	6	U	11	U	7	U	9	U	8	U
Chlorobenzene	150000	40	UJ	23	UJ	15	UJ	2	J	3	J	5	J	6	U	11	U	7	U	9	U	8	U
Chloroform	3600	40	U	23	U	15	U	11	UJ	8	U	16	U	6	U	11	U	7	U	9	U	8	U
Ethylbenzene	8900	12	J	23	UJ	15	UJ	11	UJ	8	U	16	UJ	6	U	11	U	7	U	9	U	8	U
Methylene Chloride	9100	10	J	23	U	15	U	2	J	8	U	16	U	6	U	11	U	10	UJ	10	U	11	U
Tetrachloroethene	1500	40	UJ	23	UJ	15	UJ	11	UJ	8	U	16	UJ	6	U	21		7	U	9	U	8	U
Semivolatile Organic Analysis (UG/KG)																							
Acenaphthene	3700000	690	U	700	UJ	81	J	600	UJ	510	U	540	U	370	U	440	U	490	U	530	U	440	U
Acenaphthylene	56000	680	U	700	UJ	78	J	160	J	510	U	530	U	370	U	440	U	480	U	520	U	430	U
Anthracene	22000000	680	U	700	UJ	270	J	260	J	510	U	530	U	370	U	440	U	480	U	520	U	430	U
Benzaldehyde	6100000	160	JEB	700	UJ	480	UJ	120	JEB	73	JEB	170	JEB	370	UJ	440	UJ	480	UJ	520	UJ	430	UJ
Benzo(a)anthracene	620	81	J	700	UJ	910		1400		510	U	180	J	370	U	140	J	480	UJ	150	J	130	J
Benzo(a)pyrene	62	92	J	700	UJ	900		1700		510	U	190	J	370	U	99	J	480	U	98	J	84	J
Benzo(b)fluoranthene	620	120	J	700	UJ	1200		2400		60	J	290	J	370	U	110	J	480	U	140	J	120	J
Benzo(g,h,i)perylene	2300000	94	J	700	UJ	670		1400		510	U	160	J	370	U	440	U	480	U	520	U	430	U
Benzo(k)fluoranthene	6200	680	U	700	UJ	560		1400		510	U	140	J	370	U	58	J	480	U	54	J	430	U
bis(2-Ethylhexyl)phthalate	35000	680	U	700	UJ	700		2300		510	U	530	U	370	U	440	U	520	U	520	U	430	U
Butylbenzylphthalate	12000000	680	U	700	UJ	480	U	150	J	510	U	530	U	370	U	440	U	480	U	520	U	430	U
Carbazole	24000	680	U	700	UJ	170	J	190	J	510	U	530	U	370	U	440	U	480	U	520	U	430	U
Chrysene	62000	130	J	700	UJ	1300		2300		56	J	260	J	370	U	220	J	480	U	280	J	180	J
Dibenzo(a,h)anthracene	62	680	U	700	UJ	150	J	360	J	510	U	530	U	370	U	440	U	480	U	520	U	430	U
Di-n-octylphthalate	2400000	680	U	700	UJ	480	U	130	J	510	U	530	U	370	UJ	440	UJ	480	UJ	520	UJ	430	UJ
Fluoranthene	2300000	150	J	700	UJ	2100		3300		74	J	360	J	370	U	220	J	230	J	240	J	220	J
Fluorene	2700000	680	U	700	UJ	96	J	72	J	510	U	530	U	370	U	440	U	480	U	520	U	430	U
Indeno(1,2,3-cd)pyrene	620	680	U	700	UJ	570		1200		510	U	130	J	370	U	440	U	480	U	520	U	430	U
Phenanthrene	22000000	75	J	700	UJ	1100		1200		510	U	190	J	370	U	170	J	480	U	130	J	150	J
Pyrene	2300000	140	J	700	UJ	1600		2800		76	J	380	J	38	J	270	J	320	J	400	J	260	J
Pesticide Analysis (UG/KG)																							
4,4'-DDD	2400	6.8	U	7.0	U	4.6	U	15	J	5.0	U	11		3.7	U	4.4	U	4.8	U	5.2	U	4.3	U
4,4'-DDE	1700	6.8	U	7.0	U	4.6	U	6.0	U	5.0	U	5.3	U	3.7	U	20		6.0		7.1		4.3	U
4,4'-DDT	1700	7.6		7.0	U	4.6	U	5.8	J	5.0	U	5.3	U	3.7	U	10		4.8	U	5.2	U	6.8	
Aldrin	29	24		3.6	U	2.4	U	11	J	2.6	U	2.7	U	1.9	U	2.2	U	2.5	U	2.7	U	2.2	U
alpha-Chlordane	1600	3.5	U	3.6	U	2.4	U	3.1	UJ	2.6	U	2.7	U	1.9	U	2.2	U	2.5	U	2.7	U	2.2	U
Dieldrin	30	50		23		4.6	U	6.0	U	5.0	U	5.3	U	3.7	U	4.4	U	4.8	U	5.2	U	4.3	U
Endrin Aldehyde	18000	6.8	U	7.0	U	4.6	U	6.0	UJ	5.0	U	5.3	U	3.7	U	7.3		4.8	U	5.2	U	4.3	U
gamma-Chlordane	1600	3.5	U	3.6	U	2.4	U	3.1	UJ	2.6	U	2.7	U	1.9	U	2.2	U	2.5	U	2.7	U	2.2	U
Methoxychlor	310000	35	U	36	U	24	U	31	U	26	U	27	U	19		22	U	25	UJ	98	J	22	U
PCB Analysis (UG/KG)																							
Aroclor-1016	3900	68	U	70	U	46	U	60	UJ	50	U	57	U	37	U	44	J	48	U	52	U	43	U
Aroclor-1254	220	68	U	70	U	66		120		50	U	57	U	37	U	44	U	48	U	52	U	43	U
Aroclor-1260	220	68	U	70	U	46	U	60	UJ	50	U	57	U	37	U	210	J	48	U	52	U	91	
Gasoline Range Organic Analysis (UG/KG)																							
Gasoline Range Organics		100	UJ	100	UJ	72	UJ	87	UJ	78	UJ	160	UJ	2500	U	4900	U	4200		4500	U	3700	U

Black Background - Criteria Exceeded; U - Not detected; UJ - Detection limit approximate; J - Quantitation approximate;
R - Rejected; NA - Not Analyzed; * - From dilution analysis

ANALYTICAL RESULTS - DETECTED CONTAMINANTS ONLY
SURFACE SOIL AND TEST PIT SOIL SAMPLES
CODDINGTON COVE RUBBLE FILL AREA
NEWPORT, RHODE ISLAND

Sample Number		CCRF-S-SS01-0001		CCRF-S-SS02-0001		CCRF-S-SS03-0001		CCRF-S-SS04-0001		CCRF-S-SS05-0001		CCRF-S-SS06-0001		CCRF-S-TP01-0010		CCRF-S-TP02-0010		CCRF-S-DP01		CCRF-S-TP03-0010		CCRF-S-TP04-0010	
Sample Location		SS01		SS02		SS03		SS04		SS05		SS06		TP01		TP02		TP03		TP03		TP04	
Date Sampled		7/28/2004		7/28/2004		7/28/2004		7/28/2004		7/28/2004		7/28/2004		5/18/2004		5/18/2004		5/19/2004		5/19/2004		5/19/2004	
Interval		0.0-1.0		0.0-1.0		0.0-1.0		0.0-1.0		0.0-1.0		0.0-1.0		0.0-8.0		0.0-1.0		0.0-0.0		0.0-10.0		0.0-10.0	
QC Identifier	Region 9 PRG - Residential Soil_2002	None		Field Dup. CCRF-S-SS02-0001		None		None		None		None		None		None		Field Dup. CCRF-S-TP03-0010		Field Dup. CCRF-S-TP03-0010		None	
Diesel Range Organic Analysis (MG/KG)																							
Diesel Range Organics		47		75		670		730		18	U	63		13	U	240		27		33		41	
TAL Metal Analysis (MG/KG)																							
Aluminum	76000	14500		14100		4490		11500		12700		19600		11400		55400		12400		15700		12000	
Antimony	31	0.95	UJ	1.5	UJ	0.26	UJ	0.42	UJ		R	0.56	UJ	0.15	UJ	3.2	J	0.21	UJ	0.23	UJ	0.19	UJ
Arsenic	0.39	10.5	J	9.8	J	4.1	J	16.4	J	8.9	J	16.0	J	14.8	J	6.2	J	8.1	J	11.7	J	6.9	J
Barium	5400	48.1	J	65.6	J	19.9	J	64.3	J	42.5	J	76.6	J	22.5	J	391		36.9		43.5		26.0	
Beryllium	150	1.1	J	1.1	J	0.62	J	0.97	J	1.0	J	1.5	J	0.49		0.61		0.67		0.83		0.65	
Cadmium	37	0.72	J	0.77	J	0.48	J	1.1	J	0.53	J	1.2	J	0.0097	UJ	1.3	J	0.014	UJ	0.015	UJ	0.013	UJ
Calcium		3540		4200		1430		2330		1390		3320		1820		3400		1610		1990		2110	
Chromium	210	13.9	J	10.8	J	25.4		23.3	J	14.3	J	17.9	J	15.1		31.4		13.5		16.3		12.3	
Cobalt	900	9.0	J	8.5	J	4.5	J	10.7	J	7.6	J	11.1	J	13.8	J	8.5	J	8.3	J	9.7	J	9.3	J
Copper	3100	22.7	J	28.9	J	37.6	J	74.6	J	19.1	J	49.0	J	23.6	J	716		28.9	J	31.8	J	20.7	J
Iron	23000	27500		25000		10900		30000		23300		37300		33300		18900		24100		29000		25500	
Lead	750	147	J	118	J	30.7	J	137	J	73.6	J	160	J	17.4	J	199		83.1		80.0	J	26.6	J
Magnesium		2770	J	2320	J	2970	J	3270	J	2220	J	4190	J	3670		1700		2340		2830		2720	
Manganese	1800	781	J	1890	J	231	J	364	J	415	J	2310	J	489		331		298		340		407	
Mercury	23	0.12		0.17		0.051		0.23		0.074		0.20		0.019	J	0.10	J	0.099	J	0.19	J	0.045	J
Nickel	1600	16.0	J	16.9	J	12.9	J	22.7	J	14.7	J	24.6	J	22.6	J	31.5	J	14.7	J	19.1	J	16.8	J
Potassium		350	J	280	J	348	J	529	J	224	J	524	J	482	J	461	J	319	J	430	J	297	J
Selenium	390	2.2	J	2.4	J	0.96	J	0.35	J	1.9	J	3.6	J	2.1		1.2		2.3		2.8		2.0	
Silver	390	0.19	UJ	0.19	UJ	0.14	UJ	0.15	UJ	0.15	UJ	0.30	UJ	0.097	U	57.9		0.14	U	0.15	U	0.13	U
Sodium		207	J		R		R	221	J		R	273	J		R	786	J		R		R		R
Vanadium	550	37.3	J	42.2	J	14.8	J	31.6	J	32.2	J	55.1	J	18.3		26.4		27.5		41.0		18.8	
Zinc	23000	234	J	629	J	81.8	J	248	J	119	J	177	J	62.3		4040		62.0		77.2		57.0	

Black Background - Criteria Exceeded; U - Not detected; UJ - Detection limit approximate; J - Quantitation approximate;
R - Rejected; NA - Not Analyzed; * - From dilution analysis

ANALYTICAL RESULTS - DETECTED CONTAMINANTS ONLY
SURFACE SOIL AND TEST PIT SOIL SAMPLES
CODDINGTON COVE RUBBLE FILL AREA
NEWPORT, RHODE ISLAND

Sample Number		CCRF-S-TP05-0010		CCRF-S-TP06-0608		CCRF-S-TP07-0204		CCRF-S-TP07-0608		CCRF-S-TP08-0204		CCRF-S-TP09-0204		CCRF-S-TP09-1112		CCRF-S-TP-DP02		CCRF-S-TP06-0204		CCRF-S-TP10-0204		CCRF-S-TP10-0406	
Sample Location		TP05		TP06		TP07		TP07		TP08		TP09		TP09		TP10		TP10		TP10		TP10	
Date Sampled		5/19/2004		5/19/2004		5/20/2004		5/20/2004		5/20/2004		5/20/2004		5/20/2004		5/20/2004		5/19/2004		5/20/2004		5/20/2004	
Interval		0.0-10.0		6.0-8.0		2.0-4.0		6.0-8.0		2.0-4.0		2.0-4.0		11.0-12.0		0.0-0.0		2.0-4.0		2.0-4.0		4.0-6.0	
QC Identifier	Region 9 PRG - Residential Soil_2002	None		None		None		None		None		None		None		Field Dup. CCRF-S-TP10-0406		None		None		Field Dup. CCRF-S-TP10-0406	
Volatile Organic Analysis (UG/KG)																							
1,4-Dichlorobenzene	3400	6	U	4	U	4	U	4	U	4	U	5	U	4	U	5	U	5	U	4	U	5	U
Acetone	1600000	28	JEB	12	EB	6	JEB	14	JEB	7	JEB	6	JEB	16	EB	8	JEB	1600	*JEB	8	JEB	2900	*JEB
Benzene	600	6	U	4	U	4	U	4	U	4	U	5	U	4	U	5	U	5	U	4	U	5	U
Chlorobenzene	150000	6	U	4	U	4	U	4	U	4	U	5	U	4	U	5	U	5	U	4	U	5	U
Chloroform	3600	6	U	4	U	4	U	4	U	4	U	1	J	4	U	1	J	5	U	0.9	J	1	J
Ethylbenzene	8900	6	U	4	U	4	U	4	U	4	U	5	U	4	U	5	U	5	U	4	U	5	U
Methylene Chloride	9100	8	UJ	8	U	4	UJ	5	UJ	5	UJ	9	UJ	6	U	8	UJ	17	UJ	4	UJ	8	UJ
Tetrachloroethene	1500	6	U	4	U	4	U	4	U	4	U	5	U	4	U	5	U	5	U	4	U	5	U
Semivolatile Organic Analysis (UG/KG)																							
Acenaphthene	3700000	440	U	380	U	59	J	56	J	360	U	380	U	380	U	390	U	380	U	370	U	390	U
Acenaphthylene	56000	430	U	380	U	85	J	360	U	360	U	380	U	380	U	380	U	380	U	360	U	390	U
Anthracene	22000000	430	U	380	U	170	J	130	J	360	U	380	U	380	U	120	J	38	J	360	U	390	U
Benzaldehyde	6100000	430	UJ	380	UJ	360	UJ	360	UJ	360	UJ	380	UJ	380	UJ	380	UJ	380	UJ	360	UJ	390	UJ
Benzo(a)anthracene	620	430	U	380	U	670		430		84	J	380	U	380	U	110	J	200	J	360	UJ	99	J
Benzo(a)pyrene	62	430	U	380	U	600		370		59	J	380	U	380	UJ	60	J	160	J	47	J	54	J
Benzo(b)fluoranthene	620	430	U	380	U	730		440		65	J	380	U	380	UJ	83	J	200	J	55	J	68	J
Benzo(g,h,i)perylene	2300000	430	U	380	U	400		240	J	360	U	380	U	380	U	380	U	120	J	360	U	390	U
Benzo(k)fluoranthene	6200	430	U	380	U	300	J	190	J	360	U	380	U	380	U	380	U	88	J	360	U	390	U
bis(2-Ethylhexyl)phthalate	35000	430	U	380	U	360	U	360	U	360	U	380	U	380	U	380	U	380	U	360	UJ	390	UJ
Butylbenzylphthalate	12000000	430	U	380	U	360	U	360	U	360	U	380	U	380	U	380	U	380	U	360	UJ	390	UJ
Carbazole	24000	430	U	380	U	91	J	360	U	360	U	380	U	380	U	84	J	380	U	360	U	390	U
Chrysene	62000	430	U	380	U	1100		700		150	J	380	U	380	U	180	J	340	J	360	UJ	140	J
Dibenzo(a,h)anthracene	62	430	U	380	U	120	J	360	U	360	U	380	U	380	U	380	U	380	U	360	U	390	U
Di-n-octylphthalate	2400000	430	UJ	380	UJ	360	UJ	360	UJ	360	UJ	380	UJ	380	UJ	380	UJ	380	UJ	360	UJ	390	UJ
Fluoranthene	2300000	430	U	380	U	1000		830		110	J	380	U	380	U	220	J	250	J	360	U	160	J
Fluorene	2700000	430	U	380	U	49	J	56	J	360	U	380	U	380	UJ	380	U	380	U	360	U	390	U
Indeno(1,2,3-cd)pyrene	620	430	U	380	U	320	J	220	J	360	U	380	U	380	UJ	380	U	95	J	360	U	390	U
Phenanthrene	22000000	430	U	380	U	670		560		88	J	380	U	380	UJ	150	J	120	J	360	U	120	J
Pyrene	2300000	430	U	48	J	1200	J	820	J	190	J	380	UJ	380	UJ	250	J	370	J	360	UJ	200	J
Pesticide Analysis (UG/KG)																							
4,4'-DDD	2400	4.3	U	3.7	U	3.7	U	4.2		3.6	U	3.8	U	3.8	U	3.8	U	18		3.7	U	3.9	U
4,4'-DDE	1700	4.3	U	3.7	U	11		8.3		14		3.8	U	3.8	U	3.8	U	41		3.7	U	3.9	U
4,4'-DDT	1700	4.3	U	3.7	U	4.2		6.9		37		7.4		3.8	U	3.8	UJ	42		5.6		14	J
Aldrin	29	2.2	U	1.9	U	1.9	U	1.9	U	1.8	U	1.9	U	1.9	U	2.0	U	1.9	U	1.9	U	2.0	U
alpha-Chlordane	1800	2.2	U	1.9	U	1.9	U	2.8		2.3		1.9	U	1.9	U	2.0	U	4.5		1.9	U	2.0	U
Dieldrin	30	4.3	U	3.7	U	3.7	U	3.6	U	3.6	U	3.8	U	3.8	U	3.8	U	3.8	U	3.7	U	3.9	U
Endrin Aldehyde	18000	4.3	U	3.7	U	18		8.2		3.6	U	3.8	U	3.8	U	3.8	U	3.8	U	3.7	U	3.9	U
gamma-Chlordane	1600	2.2	U	1.9	U	5.7		1.9		2.2		1.9	U	1.9	U	2.0	U	4.5		1.9	U	2.0	U
Methoxychlor	310000	22	U	19	U	19	U	19	U	18	U	19	U	19	U	20	U	19	U	19	U	20	U
PCB Analysis (UG/KG)																							
Aroclor-1016	3900	43	U	37	U	37	U	36	U	36	U	38	U	38	U	38	U	38	U	37	U	39	U
Aroclor-1254	220	43	U	37	U	37	U	36	U	36	U	38	U	38	U	38	U	38	U	37	U	39	U
Aroclor-1260	220	43	U	37	U	520		300		36	U	38	U	38	U	38	U	160		37	U	39	U
Gasoline Range Organic Analysis (UG/KG)																							
Gasoline Range Organics		4000		2000	U	2000	U	2100	U	2600	U	2300	U	2500	U	2500	U	3500	U	2200	U	2200	U

Black Background - Criteria Exceeded; U - Not detected; UJ - Detection limit approximate; J - Quantitation approximate;
R - Rejected; NA - Not Analyzed; * - From dilution analysis

ANALYTICAL RESULTS - DETE CONTAMINANTS ONLY
SURFACE SOIL AND TES PIT SOIL SAMPLES
CODDINGTON COVE RUBBLE FILL AREA
NEWPORT, RHODE ISLAND

Sample Number		CCRF-S-TP05-0010		CCRF-S-TP06-0608		CCRF-S-TP07-0204		CCRF-S-TP07-0608		CCRF-S-TP08-0204		CCRF-S-TP09-0204		CCRF-S-TP09-1112		CCRF-S-TP-DP02		CCRF-S-TP06-0204		CCRF-S-TP10-0204		CCRF-S-TP10-0406	
Sample Location		TP05		TP06		TP07		TP07		TP08		TP09		TP09		TP10		TP10		TP10		TP10	
Date Sampled		5/19/2004		5/19/2004		5/20/2004		5/20/2004		5/20/2004		5/20/2004		5/20/2004		5/20/2004		5/19/2004		5/20/2004		5/20/2004	
Interval		0.0-10.0		6.0-8.0		2.0-4.0		6.0-8.0		2.0-4.0		2.0-4.0		11.0-12.0		0.0-0.0		2.0-4.0		2.0-4.0		4.0-6.0	
QC Identifier	Region 9 PRG - Residential Soil_2002	None		None		None		None		None		None		None		Field Dup. CCRF-S-TP10-0406		None		None		Field Dup. CCRF-S-TP10-0406	
Diesel Range Organic Analysis (MG/KG)																							
Diesel Range Organics		15		220		210		160		35		22		14	U	28	J	150		51		68	J
TAL Metal Analysis (MG/KG)																							
Aluminum	76000	12600		14800		9830		8410		8300		10700		10400		13100		10800		11200		11800	
Antimony	31	0.19	UJ	0.16	UJ	0.16	UJ	0.16	UJ	0.14	UJ	4.5	J	0.15	UJ	0.17	UJ	0.17	UJ	0.16	UJ	0.17	UJ
Arsenic	0.39	9.2	J	9.1	J	17.7	J	9.9	J	6.9	J	6.4	J	6.7	J	9.6	J	10.2	J	11.6	J	9.2	J
Barium	5400	25.9		12.8	J	30.6		22.4		16.8	J	19.7	J	18.9	J	27.5		23.2		22.1	J	23.7	J
Beryllium	150	0.80		0.48		0.47		0.45		0.47		0.52		0.51		0.67		0.59		0.55		0.57	
Cadmium	37	0.013	UJ	0.011	UJ	2.6	J	0.41	J	0.0095	UJ	0.010	UJ	0.010	UJ	0.011	UJ	0.011	UJ	0.011	UJ	0.011	UJ
Calcium		955		1740		2400		3460		1200		1620		558		1500		2880		2100		1290	
Chromium	210	12.4		18.0		17.6		11.1		8.6		16.7		12.9		12.5		11.2		12.6		12.3	
Cobalt	900	9.3	J	16.1	J	17.2	J	10.3	J	7.1	J	12.9	J	6.7	J	9.8	J	9.0	J	10.5	J	10.1	J
Copper	3100	14.2	J	32.3	J	80.5	J	33.0	J	16.1	J	27.4	J	16.2	J	15.7	J	21.6	J	20.2	J	18.2	J
Iron	23000	21700		39800		24500		19600		18000		29800		21300		24700		20600		29200		25500	
Lead	750	16.6	J	32.8	J	55.7	J	44.0	J	34.9	J	33.9	J	9.3	J	30.9	J	20.5	J	26.8	J	31.1	J
Magnesium		2400		5140		3180		2490		2080		3210		2980		2740		3090		3040		3000	
Manganese	1800	277		612		313		262		274		410		232		462		289		403		392	
Mercury	23	0.047	J	0.032	J	0.077	J	0.052	J	0.046	J	0.066	J	0.022	J	0.072	J	0.070	J	0.051	J	0.045	J
Nickel	1600	14.0	J	28.6	J	21.7	J	14.0	J	13.2	J	22.9	J	13.8	J	17.5	J	14.1	J	17.6	J	18.1	J
Potassium		312	J	320	J	445	J	410	J	306	J	271	J	297	J	274	J	429	J	285	J	276	J
Selenium	390	2.0		2.5		1.4		1.4		1.3		1.9		1.8		2.3		2.2		2.5		2.0	
Silver	390	0.13	U	0.11	U	0.10	U	0.11	U	0.095	U	0.10	U	0.10	U	0.11	U	0.11	U	0.11	U	0.11	U
Sodium			R		R		R		R		R		R		R		R		R		R		R
Vanadium	550	21.6		18.1		21.7		15.7		14.7		16.5		21.1		20.2		16.8		18.7		20.0	
Zinc	23000	40.3		64.0		112		81.4		53.3		84.0		39.0		57.4		52.3		56.3		56.9	

Black Background - Criteria Exceeded; U - Not detected; UJ - Detection limit approximate; J - Quantitation approximate;
R - Rejected; NA - Not Analyzed; * - From dilution analysis

ANALYTICAL RESULTS - DE[illegible]ED COMPOUNDS ONLY
QA/QC SAMPLES
CODDINGTON COVE RUBBLE FILL AREA
NEWPORT, RHODE ISLAND

Sample Number		CCRF-A-SS03-0001-RB01		CCRF-A-DIUF		CCRF-A-TB01		CCRF-A-TB02		CCRF-A-TB03		CCRF-A-TB04		CCRF-A-TP10-0406-RB01	
Sample Location		Blank		Blank		Blank		Blank		Blank		Blank		TP01	
Date Sampled		7/28/2004		5/20/2004		5/18/2004		5/19/2004		5/20/2004		7/28/2004		5/20/2004	
QC Identifier	MCL	Rinsate Blank		Source Blank		Trip Blank		Trip Blank		Trip Blank		Trip Blank		Rinsate Blank	
Volatile Organic Analysis (UG/L)															
2-Butanone		5	U	3	J	5	U	5	U	5	U	5	U	5	U
Acetone		30	J	10	J	5	U	5	U	2	J	5	UJ	14	J
Semivolatile Organic Analysis (UG/L)															
Benzaldehyde		2	J	10	UJ		NA		NA		NA		NA	10	UJ
bis(2-Ethylhexyl)phthalate	6	2	J	18			NA		NA		NA		NA	22	
Phenol		10	U	3	J		NA		NA		NA		NA	2	J
TAL Metal Analysis (UG/L)															
Calcium		107	J	74.0	U		NA		NA		NA		NA	114	UJ
Lead	15	2.0	U	2.2	J		NA		NA		NA		NA	2.0	U

Black Background - Criteria Exceeded; U - Not detected; UJ - Detection limit approximate; J - Quantitation approximate;
R - Rejected; NA - Not Analyzed; * - From dilution analysis

TETRA TECH NUS, INC.
55 Jonspin Road • Wilmington, MA 01887-1020
Tel 978.658.7899 • Fax 978.658.7870 • www.tetratech.com

C-NAVY-08-04-1738W

Date: July 9, 2004 c: File N5152-D-4.10

To: Steve Parker

From: Dan Wielandt

Subject: Tier II Data Validation Organic/Inorganic Data
Mitkem Corporation Project Number C0442
CTO-842, Coddington Cove Rubble Fill Area

VOC/SVOC/Pesticide/PCB/GRO/DRO/Metals:
16/Soils/ CCRF-S-DP01, CCRF-S-TPO1-0008, CCRF-S-TP02-0001, CCRF-S-TP03-0010, CCRF-S-TP04-0010, CCRF-S-TP05-0010, CCRF-S-TP06-0204, CCRF-S-TP06-0608, CCRF-S-TP07-0204, CCRF-S-TP07-0608, CCRF-S-TP08-0204, CCRF-TP09-0204, CCRF-S-TP09-1112, CCRF-S-TP10-0204, CCRF-S-TP10-0406, CCRF-S-TP-DP02
(Field Duplicate Pairs: CCRF-S-TP03-0010/CCRF-S-DP01 and CCRF-S-TP10-0406/CCRF-S-TP-DP02)

VOC/SVOC/Pesticide/PCB/GRO/DRO/Metals:
7/Groundwaters/ CCRF-A-DP01, CCRF-GW-TP01, CCRF-GW-TP02, CCRF-GW-TP03, CCRF-GW-TP04, CCRF-GW-TP05, CCRF-GW-TP08
(Field Duplicate Pair: CCRF-GW-TP04/CCRF-A-DP01)

1/Source Blank/ CCRF-A-DIUF

1/Rinsate Blank/ CCRF-A-TP10-0406-RB01

VOC:
2/Trip Blanks/ CCRF-A-TB01, CCRF-A-TB02

VOC/GRO:
1/Trip Blank/ CCRF-A-TB03

A Tier II data validation was performed by Tetra Tech NUS, Inc. (TtNUS) on the volatile (VOC), semivolatile (SVOC), pesticide, polychlorinated biphenyl compounds (PCB), gasoline range organics (GRO), diesel range organics (DRO), and metals data from the soil and groundwater samples collected at the Coddington Cove Rubble Fill Area on May 18, 19, and 20, 2004. The VOC and SVOC analyses were performed according to USEPA SW-846 Methods 8260B and 8270C, respectively. The pesticide and PCB analyses were performed according to USEPA SW-846 Method 8081 and 8082, respectively. The GRO and DRO analyses were performed according to Modified USEPA SW-846 Method 8015B. The metals analysis was performed according to SW-846 Method 6010B/7470A. The volatile, semivolatile, GRO, and DRO data validation was performed in accordance with the Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, December 1996. . The pesticide and PCB data validation was performed according to the Region I, EPA Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses, November 1988.

The metals data validation was performed using the Region I, EPA-NE Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, modified February 1989.

ORGANIC DATA

The data were evaluated based on the following parameters:

	• Overall Evaluation of Data and Potential Usability Issues
*	• Data Completeness (CSF Audit - Tier I)
*	• Preservation and Technical Holding Times
*	• GC/MS Instrument Performance Check (Tuning)
	• Initial and Continuing Calibrations
	• Blanks
	• Surrogate Compounds
	• Internal Standards
	• Matrix Spike/Matrix Spike Duplicate
	• Laboratory Control Sample/Laboratory Control Sample Duplicate
	• Field Duplicates
NA	• Target Compound Identification
NA	• Compound Quantitation and Reported Quantitation Limits
*	• Tentatively Identified Compounds
*	• System Performance

NA – Parameter not included in a Tier II level data validation.

* All criteria were met for this parameter.

Initial and Continuing Calibrations

Volatiles

The following table summarizes the volatile compounds that failed to meet the continuing calibration (CC) criterion of %D < 25:

Compound	Action (+)	Action NDs	Affected Samples
Dichlorodifluoromethane		UJ	S-TP02-0001, S-TP04-0010
1,1,2-Trichloro-1,2,2-trifluoromethane		UJ	
2-Butanone		UJ	S-TP05-0010
4-Methyl-2-pentanone		UJ	
2-Hexanone		UJ	

Compound	Action		Affected Samples
	(+)	NDs	
Acetone	J		S-TP06-0204, S-TP10-0406, A-DP01, A-TB03, A-TP10-0406-RB01, A-DIUF, GW-TP08
Carbon Disulfide		UJ	A-DP01, A-TB03, A-TP10-0406-RB01, A-DIUF, GW-TP08
2-Hexanone		UJ	

Semivolatiles

The following table summarizes the semivolatile compounds that failed to meet the initial calibration (IC) criterion of %RSD < 30 and the continuing calibration (CC) criterion of %D < 25:

Compound	Action		Affected Samples
	(+)	NDs	
2,4-Dinitrophenol		UJ	All soil and groundwater samples
3,3'-dichlorbenzidine		UJ	
Benzaldehyde		UJ	
Pyrene	J	UJ	S-TP09-1112, S-TP09-0204, S-TP-DP02, S-TP08-0204, S-TP10-0204, S-TP10-0406, S-TP02-0001, S-TP07-0204, S-TP07-0608
4-Chloroaniline		UJ	GW-TP01, GW-TP02, GW-TP03, GW-TP04, GW-TP05, A-DP01, A-TP10-0406-RB01, A-DIUF, GW-TP08
Atrazine		UJ	
Benzo(a)anthracene		UJ	S-DP01
Atrazine		UJ	

Blanks

Volatiles

The following table summarizes the level of blank contamination detected in the field and laboratory blanks associated with these samples

Compound	Type of Blank	Maximum Concentration	Action Level	Affected Samples
Methylene Chloride	Method	7 ug/Kg	70 ug/Kg	All soil samples
Acetone	Trip	2 ug/L	20 ug/L	A-DP01, GW-TP04, GW-TP05, GW-TP08
Acetone	Rinsate	14 ug/L	140 ug/L	S-DP01, S-TP03-0010, CCRFS-TP04-0010, S-TP05-0010, S-TP06-0204, S-TP06-0608, S-TP07-0204, S-TP07-0608, S-TP08-0204, TP09-0204, S-TP09-1112, S-TP10-0204, S-TP10-0406, S-TP-DP02

The positive methylene chloride results are reported as non-detected (U) in all soil samples. The positive acetone results in the affected groundwater samples are reported as non-detected (U). Since methylene chloride and acetone are common laboratory contaminants, the 10x rule applies for action limits. The data may be biased high or false positive. The positive acetone results in the soil samples are qualified (EB) to indicate that the acetone results may be due to contamination form the rinsate blank. These positive acetone results may be false positive or biased high.

Semivolatiles

The following table summarizes the level of blank contamination detected in the field and laboratory blanks associated with these samples.

Compound	Type of Blank	Maximum Concentration	Action Level	Affected Samples
bis(2-Ethylhexyl)phthalate	Rinsate	22 ug/L	220 ug/L	A-DP01, GW-TP01, GW-TP02, GW-TP03, GW-TP04, GW-TP05, GW-TP08
bis(2-Ethylhexyl)phthalate	Method	370 ug/Kg	3700 ug/Kg	S-DP01, S-TPO1-0008, S-TP03-0010, S-TP04-0010, S-TP05-0010, S-TP06-0608, S-TP07-06-08, S-TP08-0204, S-TP09-1112, S-TP10-0204

Blank actions were applied to the selected soil and groundwater samples due to bis(2-ethylhexyl)phthalate blank contamination. Since bis(2-ethylhexyl)phthalate is a common laboratory contaminants, the 10x rule applies for action limits. The data may be biased high or false positive.

The following actions apply for blank contamination:

- Accept values > Action Level.
- Report as (U) values > CRQL and < Action Level.
- Report CRQL (U) values < CRQL and < Action Level.

Surrogate Recovery

Volatiles

The percent recovery for 1,2-dichloroethane-d_4 was above the quality control limits (50 - 126) for samples S-DP01, S-TP06-0204, S-TP07-0204, S-TP07-0608, S-TP08-0204, S-TP09-1112, S-TP10-0204, S-TP10-0406, S-TP-DP02, and S-TP05-0010. The positive results are estimated (J) in the affected samples. The results may be biased high.

Internal Standards

Semivolatiles

The following table summarizes the internal standard areas outside the quality control limits.

Internal Standard	IS Area	Acceptable Range	Action		Affected Sample
			(+)	NDs	
Chrysene-d_{12}	597189	599128 – 2396510		UJ	S-TP10-0204
Acenaphthene-d_{10}	413341	420183 – 1680732		UJ	GW-TP05
Acenaphthene-d_{10}	402071	420183 – 1680732		UJ	GW-TP08
Phenanthrene-d_{10}	686833	689818 – 2759270		UJ	

The compounds associated with internal standards outside quality control limits are estimated (UJ) in the affected soil and groundwater samples. The bias is undetermined.

Matrix Spike/Matrix Spike Duplicate

Semivolatiles

The following tables summarize the semivolatile matrix spiking recoveries that did not meet QC limits in the matrix spike and matrix spike duplicate analysis:

GW-TP08							
Compound	MS %REC	MSD %REC	QC Limits	RPD	QC Limits	Action	
						(+)	ND
3,3'-Dichlorobenzidine	8	2	41 – 117	120	40		UJ
Di-n-octylphthalate		56	59 – 139				UJ
Benzo(a)pyrene		56	61 – 121				UJ

S-TP06-0204					
Compound	MS %REC	MSD %REC	QC Limits	Action	
				(+)	ND
2,4-Dinitrophenol	0	0	15 – 136		R
4,6-Dinitro-2-methylphenol	14	10	33 – 130		UJ
Di-n-octylphthalate	53	49	69 – 137		UJ
Benzo(b)fluoranthene	58	53	61 - 129	J	
Benzo(k)fluoranthene	58	58	62 – 130	J	
Benzo(a)pyrene	60	60	66 – 119	J	
2,4-Dinitrotoluene		58	59 – 123		UJ
Di-n-butylphthalate		68	70 –120		UJ

The non-detected result for 2,4-Dinitrophenol in sample S-TP06-0204 is rejected (R) due to 0% matrix spike recovery. The result may be false negative.

S-TP09-1112					
Compound	MS %REC	MSD %REC	QC Limits	Action	
				(+)	ND
Di-n-octylphthalate	58	58	69 – 137		UJ
Benzo(b)fluoranthene	58	58	61 – 129		UJ
Benzo(a)pyrene	58	58	66 – 119		UJ
Fluorene		58	63 – 115		UJ
Phenanthrene		68	70 – 115		UJ
Indeno(1,2,3-cd)pyrene		53	56 – 128		UJ

PCBs

The following table summarizes the PCB matrix spiking recoveries that did not meet QC limits in the matrix spike and matrix spike duplicate analysis:

S-TP02-0001					
Compound	MS %REC	MSD %REC	QC Limits	Action	
				(+)	ND
Aroclor 1016	61		62 – 155		UJ
Aroclor 1260	50	39	56 – 173	J	

Laboratory Control Sample

Semivolatiles

The following table summarizes the semivolatile spiking recoveries that did not meet QC limits in the laboratory control sample:

Compound	LCS % Recovery	QC Limits
2,4-Dinitrophenol	10	15 – 136
Di-n-octylphthalate	65	69 - 137

The non-detected results for 2,4-dinitrophenol and di-n-octylphthalate in all soil samples are estimated (UJ) due to low spike recovery. The results may be false negative.

Field Duplicates

DRO

The positive DRO results in the field duplicate pair S-TP10-0406/S-TP-DP02 are estimated (J) due to poor field duplicate precision. The bias is undetermined.

Pesticides

The positive and non-detected results for methoxychlor in the field duplicate pair S-TP03-0010/S-DP01 are estimated (J, UJ) due to poor field duplicate precision. The bias is undetermined.

The positive and non-detected results for 4,4'-DDT in the field duplicate pair S-TP10-0406/S-TP-DP02 are estimated (J) due to poor field duplicate precision. The bias is undetermined.

System Performance

The volatile, semivolatile, pesticide, PCB, GRO, DRO, and metals sample results do not indicate any major analytical data quality trends or problems.

METALS DATA

The metals data were evaluated based on the following parameters:

- Data Completeness
- Holding Times *
- Calibration Verification *
- Field and Laboratory Blank Analyses
- ICP Interference Check Sample Results
- Matrix Spike Recoveries

*	•	Laboratory Control Sample Results
	•	Laboratory Duplicate Results
*	•	Field Duplicate Precision
	•	ICP Serial Dilution Results
*	•	Detection Limits
NA	•	Sample Quantitation

NA – Parameter not evaluated for Tier II level data validation.

* - All quality control criteria were met for this parameter.

Data Completeness

The laboratory was contacted on August 5, 2004 about an error in the Form VI (Duplicates) and about missing raw data for the calculation of percent solids. The missing percent solids raw data was received by fax on August 5, 2004, and the corrected Form VI was received on August 6, 2004. The laboratory indicated that the corrected electronic deliverables would be sent shortly.

The ICP serial dilution analysis for soil samples was only reported for barium. Therefore, the soils data for metals other than barium are not evaluated based on this parameter.

Field and Laboratory Blank Analyses

The laboratory blank analyses were used to calculate the maximum concentrations and action levels of the following contaminants affecting the groundwater samples:

Analyte	Maximum Concentration (µg/L)	Action Level (µg/L)
Antimony	6.7	33.5
Beryllium	0.5	2.5
Copper	12.2	61.0
Nickel	1.9	9.5
Silver	3.2	16.0
Thallium	5.0	25.0
Vanadium	3.1	15.5

The positive results below the action level for antimony, beryllium, copper, nickel, silver, thallium, and vanadium were changed to non-detected values in the groundwater samples due to laboratory blank contamination.

The laboratory and rinsate blank analyses were used to calculate the maximum concentrations and action levels of the following contaminants affecting the soil samples:

Analyte	Maximum Concentration (µg/L)	Action Level (mg/kg)
Sodium	408	102
Thallium	5.0	1.3

The positive results below the action level for sodium and thallium were changed to non-detected values in the soil samples due to laboratory blank contamination.

The action levels in the table above are based on an assumption of 100 percent solids, 50 ml of final volume, and 1 gram of sample analyzed. The action level for each soil sample is different, based on the actual percent solids and amount of soil sample analyzed.

The laboratory blank analyses were used to calculate the maximum concentrations and action levels of the following contaminants affecting the field blank samples:

Analyte	Maximum Concentration (µg/L)	Action Level (µg/L)
Aluminum	75.4	377
Barium	8.7	43.5
Cadmium	0.83	4.2
Calcium	459	2295
Copper	12.2	61.0
Iron	54.8	274
Manganese	3.8	19.0
Sodium	447	2235
Zinc	2.1	10.5

The positive results below the action level for aluminum, barium, cadmium, calcium, copper, iron, manganese, sodium, and zinc were changed to non-detected values in the field blank samples due to laboratory blank contamination.

ICP Interference Check Sample Results

The following results were qualified in the affected samples due to ICP interference due to the high concentrations of iron in all soil samples and aluminum in samples S-TP02-0001 and S-TP06-0608:

Analyte	Action	Affected samples
Arsenic	J	All soil samples
Barium	J	S-TP01-0008, S-TP06-0608, S-TP08-0204, S-TP09-0204, S-TP09-1112, S-TP10-0204, S-TP10-0406
Cadmium	J+*, UJ-NDs*	All soil samples
Cobalt	J	All soil samples
Copper	J*	S-DP01, S-TP01-0008, S-TP03-0010, S-TP04-0010, S-TP05-0010, S-TP06-0204, S-TP06-0608, S-TP07-0204, S-TP07-0608, S-TP08-0204, S-TP09-0204, S-TP09-1112, S-TP10-0204, S-TP10-0406, S-TP-DP02
Lead	J	S-TP01-0008, S-TP03-0010, S-TP04-0010, S-TP05-0010, S-TP06-0204, S-TP06-0608, S-TP07-0204, S-TP07-0608, S-TP08-0204, S-TP09-0204, S-TP09-1112, S-TP10-0204, S-TP10-0406, S-TP-DP02
Nickel	J	All soil samples
Potassium	J	All soil samples

Analyte	Action	Affected samples
Sodium	R	S-DP01, S-TP01-0008, S-TP03-0010, S-TP04-0010, S-TP05-0010, S-TP06-0204, S-TP06-0608, S-TP07-0204, S-TP07-0608, S-TP08-0204, S-TP09-0204, S-TP09-1112, S-TP10-0204, S-TP10-0406, S-TP-DP02
	J	S-TP02-0001

* Results are biased low. No * indicates results biased high.

The cadmium and copper results are estimated due to negative ICP interference from iron and/or aluminum in the affected samples. Results may be biased low or false negative.

The results for arsenic, barium, cobalt, lead, nickel, potassium, and sodium are estimated due to positive ICP interference from iron and/or aluminum in the affected samples. The results may be biased high. All results for sodium except in sample S-TP02-0001 are rejected because the results may be entirely due to positive ICP interference from iron and/or aluminum. The results may be false positive.

Matrix Spike Recoveries

The positive and non-detected antimony results are estimated (J, UJ) in the soil samples due to the low matrix spike recovery. The results may be biased low.

Laboratory Duplicate Results

The positive mercury results are estimated (J) in all soil samples due to poor laboratory duplicate precision. The bias is undetermined.

ICP Serial Dilution Results

The positive barium, nickel, and vanadium results are estimated (J) in all of the groundwater samples due to a sample matrix suppressing effect detected in the ICP serial dilution analysis. The results may be biased low.

Overall Assessment of the Metals Data

The metals data are acceptable for use as qualified. Positive sample results changed to non-detected values due to blank contamination are treated as positive results for qualification purposes. The estimation of these non-detected values is reported in the data summary table as (UJ).

Blank actions were taken for antimony, beryllium, copper, nickel, silver, thallium, and vanadium in affected groundwater samples, and for sodium and thallium in affected soil samples, due to laboratory blank contamination. Blank actions were also taken for aluminum, barium, cadmium, calcium, copper, iron, manganese, sodium, and zinc in affected field blank samples due to laboratory blank contamination.

The cadmium and copper results are estimated due to negative ICP interference from iron and/or aluminum in the affected soil samples. Results may be biased low or false negative. The results for arsenic, barium, cobalt, lead, nickel, potassium, and sodium are estimated due to positive ICP interference from iron and/or aluminum in the affected soil samples. These results may be biased high.

All results for sodium in the soil samples except in sample S-TP02-0001 are rejected because the results may be entirely due to positive ICP interference from iron and/or aluminum. The results may be false positive.

The positive and non-detected antimony results are estimated (J, UJ) in the soil samples due to the low matrix spike recovery. The results may be biased low.

The positive mercury results are estimated (J) in all soil samples due to poor laboratory duplicate precision. The bias is undetermined.

The positive barium, nickel, and vanadium results are estimated (J) in all of the groundwater samples due to a sample matrix suppressing effect detected in the ICP serial dilution analysis. The results may be biased low.

Sample results less than 2x IDL and less than the CRDL are estimated (J) due to uncertainty in values near the instrument detection limit.

Tables: Table III: TIC Summary Table
Data Summary Tables

Enclosures: Data Validation Worksheets

APPENDIX G

PA INVESTIGATION TEST PIT LOG SHEETS

TETRA TECH NUS, INC.

JAR HEADSPACE ANALYSIS LOG

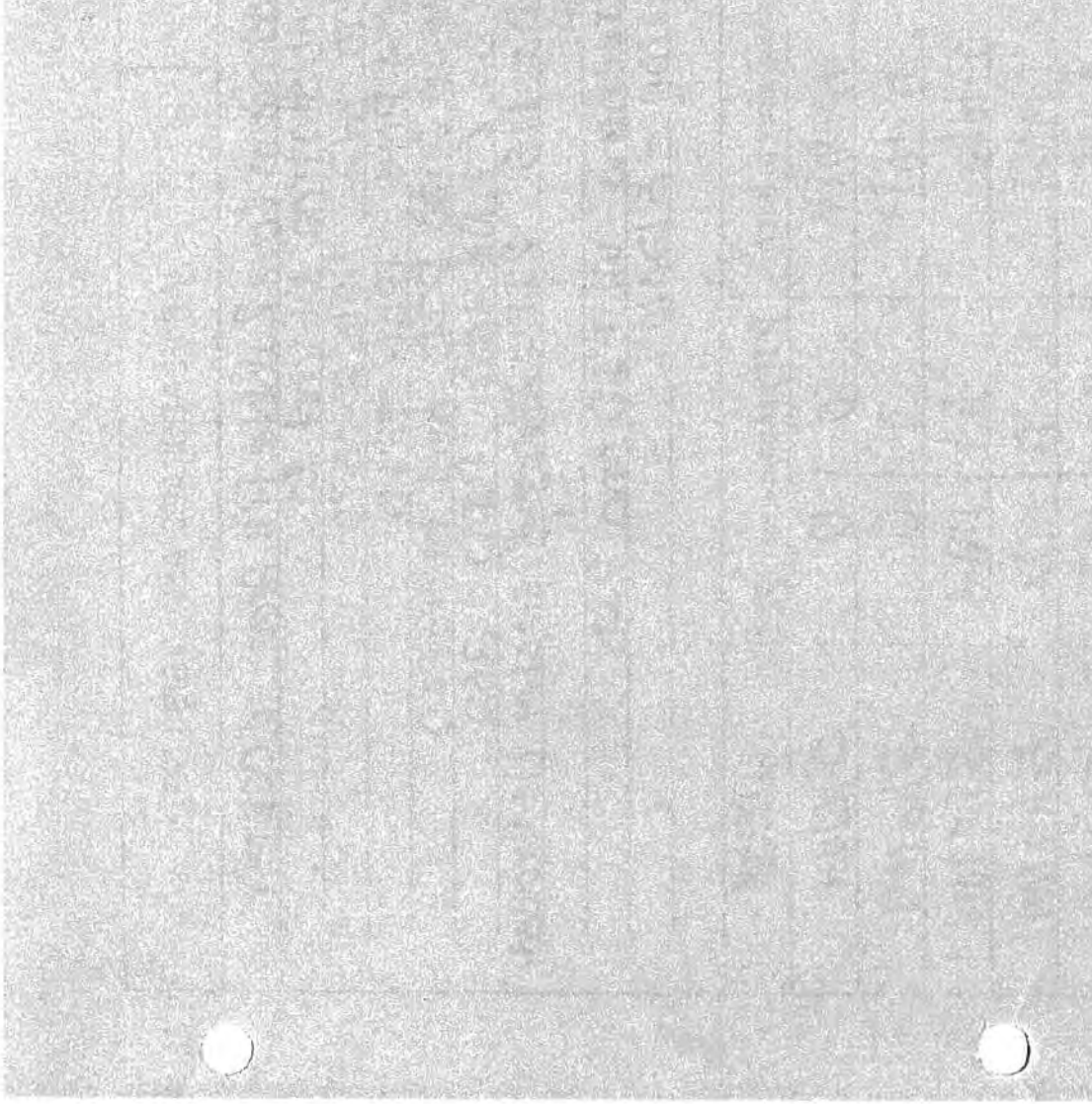
SITE NAME: Coddington Cove Rubble Fill Area (CCRF)
SITE LOCATION: Naval Station Newport RI
PROJECT NO./CTO NO.: CTO - 842 N5152-0570
SAMPLE LOCATION: Test Pits : TP01 - TP10
INSTRUMENT: Photovac 2020 PID and microFID
SERIAL NO.: FID = C2NK350 PID = 202
MODEL NO.: FID = 1/S PID = ~~2020~~ 10 5/20/04 Not Recorded.
SAMPLE PREP METHOD[1]: NA.
HEADSPACE ANALYST: Screening W/ FID and PID
DATE: 5/18/04 - 5/20/04

SAMPLE TYPE[2]/NUMBER	SAMPLE DEPTH (FEET)	PID READING (ppm)	FID	~~COMMENTS~~ Date
TP01	0-8ft	0	0	5/18/04
TP02	0-10ft	0	0	5/18/04
TP03	0-10ft	6.1	112.8	5/19/04
TP04	0-10ft	0	172	5/19/04
TP05	0-10ft	18.1	153	5/19/04
TP06	0-11ft	1.8	12.6	5/19/04
TP07	0-10ft	1.6	7.1	5/20/04
TP08	0-10ft	2.0	2.1	5/20/04
TP09	0-12ft	0.0	182	5/20/04
TP10	0-10ft	0.0	0.2	5/20/04

US Form 0008

1) (a) ambient temp
(h) heated (air)
(w) hot water bath

2) Type of Sample
SB Soil Boring
SD Sediment Sample
GW Groundwater Sample
TP Test Pit Sample
SS Soil Sample



TETRA TECH NUS, INC.

TEST PIT LOG

Site Name: CCRF Coddington Cove Rubble Fill Area (location of Proposed TP08) Test Pit No.: TP01

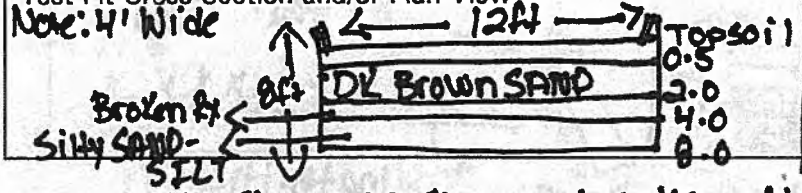
Project Number: N5152-0570 Date: 5/18/04

Location: Newport Naval Station Newport, RI Field Geologist: J. Danieli

DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Topsoil	0-0.5 - Topsoil/loam/roots	Fill	
2	Fill	0.5-2' - DK Brown SAND w/ Gravel (Sand F-C)		
4	Fill	2-4 DK Gray - layer of Broken Rx - Phy life?		
6	Native ~~materials~~ Soils	4-8' - Olive Gray - DK Gray dense/compact Fine Silty SAND - SILT w/ trace Gravels - orange staining noted.	Sm/ML	Native soils ~~material~~ @ 4' bgs
8		Bottom of Pit 8' bgs		GW ≅ 8' bgs

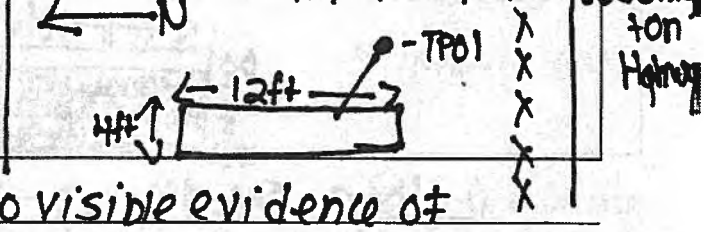
Test Pit Cross Section and/or Plan View

Note: 4' Wide



Cross section:

map view:



REMARKS: No Sheen or Stained soils - No visible evidence of Contaminated soils. Stop digging at depth of Groundwater ≅ 8' bgs Collect Soil sample - CCRF-S-TP01-0008 at 1044. Sample From entire Pit Soil scraped From Walls of pit. Collect GW sample CCRF-~~TP01~~-GW-TP01 at 1254.

PHOTO LOG: GW Silty and murky, no odor, slight sheen. 3 Photos to West (west wall of pit), 2 photos to South (South wall of pit + down into pit), 2 photos to north (north wall of pit and down into pit).

TEST PIT: TP01

PAGE 1 OF 1

Tt NUS FORM 0011

Note: Bz - 0 ppm w/ PID + FID
Headspace - 0 ppm PID + FID

TETRA TECH NUS, INC.

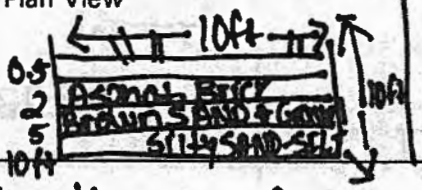
TEST PIT LOG

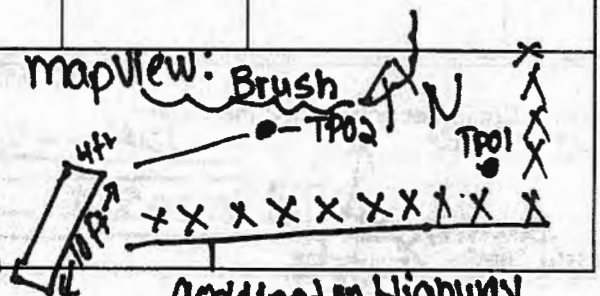
Site Name: CCRF
Project Number: N5152-0570
Location: Newport Naval Station Newport RI
Test Pit No.: TP02 (Approximate Location of Proposed TP09)
Date: 5/18/04
Field Geologist: J. Danieli

DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Topsoil	0-0.5 - Topsoil / loam / grass / roots	Fill	
2	Fill 0.5	0.5 - OK Gray M-C SAND w/ Asphalt, brick, plastic, White material (ASH?)		
4	Fill	2-5 - Brown SAND w/ Gravel		
6		5' sm/mL		Native soils ~5' bgs
8	Native materials soils	Brown-olive dense/compact Silty SAND - SILT trace Gravel, Orange staining		
10		Bottom of Pit ≅ 10' bgs		TD ≅ 10' bgs

Test Pit Cross Section and/or Plan View

Note: Width = 4'





REMARKS: 0-1' bgs - Stop digging - Asphalt, brick, white material (ASH?) - Collect soil sample - CCRF-S-TP02-0001 @ 1410
Continue to Dig to depth of GW at ~10' bgs - Collect GW Sample - CCRF-GW-TP02 @ 1515. No visible evidence of sheen or stained soils. GW no sheen, silty + turbid. no odor.

PHOTO LOG: 2 photos of excavator bucket w/ 0-1' bgs soils, 2 photos - 0-1ft bgs pit, 1-Photo to NE, 1 photo of NE + East Wall, 1 photo SW + East wall, 1 photo east wall, 1 close up of 0-1ft bgs of NE wall, 3 photos to SW.

TEST PIT: TP02

PAGE 1 OF 1

Tt NUS FORM 0011

B2 = 0.0 ppm PID + FID
Headspace = 0.0 ppm FID + PID.

TETRA TECH NUS, INC.

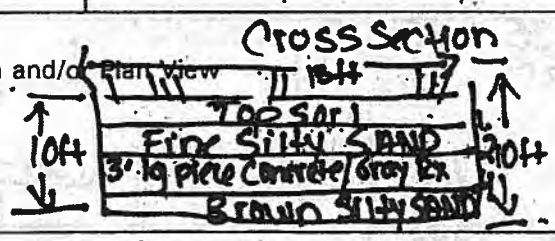
TEST PIT LOG

Site Name: CCRF
Test Pit No.: TP03 (~Location of proposed TP07)
Project Number: N5152-0570
Date: 5/19/04
Location: Naval Station Newport RI.
Field Geologist: J Daniell

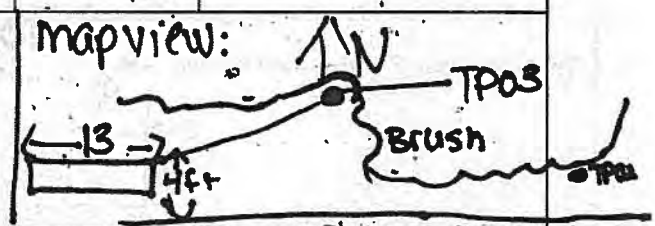
DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Topsoil	0-1 Topsoil/Loam/Grass/Roots	Fill	
2	F. Silty SAND	1-2 - Fine Silty SAND, (Brown) pieces of Gravel and concrete		
4	Concrete Broken Rock	3' - large piece of concrete 2-4' - Gray weathered Rx-Phyllite?		
6	Brown Silty	4-9' - Brown silty SAND w/		
8	SAND-w/ broken Rock	pieces of broken Rx (Phyllite) and Gravels		
10	Brown Silty SAND	9-10 Brown Silty SAND ↓ ID 5/19/04	SM	∇ ≅ 10' bgs
	Native ~~materials~~ soils	Bottom of Pit ≅ 10' bgs		Natural ~~materials~~ soils ~9' bgs

Test Pit Cross Section and/or Plan View

Note: Width = 4ft.



Map view:



REMARKS: No evidence of contaminated soils - no sheen or staining.
Soil Sample 0-10' From Walls of Pit - CCRF-S-TP030010 @ 0830 and DP01 - @ 0835 CCRF-S-DP01. Collect GW Sample - CCRF-GW-TP03 @ 0930 - GW silty and murky, no sheen + no odor.

PHOTO LOG:
1 Photo to SW, 1 photo of West + South ~~west~~ wall, 1 photo of West wall, 1 photo of North wall, 2 photos of South wall.

TEST PIT: TP03

PAGE 1 OF 1

Tt NUS FORM 0011

Headspace: FID = 112 ppm
PID = 6.1 ppm

TETRA TECH NUS, INC.

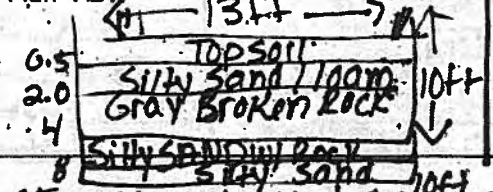
TEST PIT LOG

Site Name: CCRF-
Project Number: N5152-0570
Location: Newport Naval Station Newport, RI.
Test Pit No.: TP04 (~location of proposed TP06)
Date: 5/19/04
Field Geologist: J. Danieli

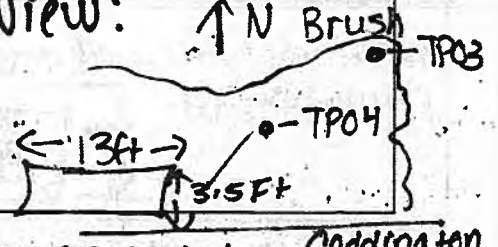
DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Topsoil	0-0.5 - Topsoil/Grass/Roots	Fill	
2	Fill/loam	0.5-2.0 - Brown Silty SAND. loam-trace roots at 2' lg piece of concrete		
4	Fill 2-4'	Gray-broken pieces of rock - phyllite? w/ layers of Brown Silty SAND w/-Gray Broken Rock.		
6				
8	Fill 4-8'			Native soils ≅ 8' bgs
10	Native materials soils	Dark Brown Sandy SILT-Silty SAND.	SM/ML	∇ ≅ 10' bgs

Test Pit Cross Section and/or Plan View

Cross Section



map view:



REMARKS: No evidence of contaminated soils - no sheen or staining
Collect Soil Sample from entire Pit - Stop at depth of GW at ~10' bgs. Soil sample - from Walls of Pit: CCRF-S-TP04-0010@1030. Collect GW Sample: CCRF-GW-TP04@1110 and GW DP01: CCRF-GW-DP01 at 1120

PHOTO LOG: GW turbid, silty and no sheen and no odor.
Photo 1 west wall, Photo 2 South wall, Photo 3 east wall, Photos 4 and 5 North walls.

TEST PIT: TP04

PAGE 1 OF 1

Tt NUS FORM 0011

Headspace: FID= 172.0 ppm
PID= 0.0 ppm

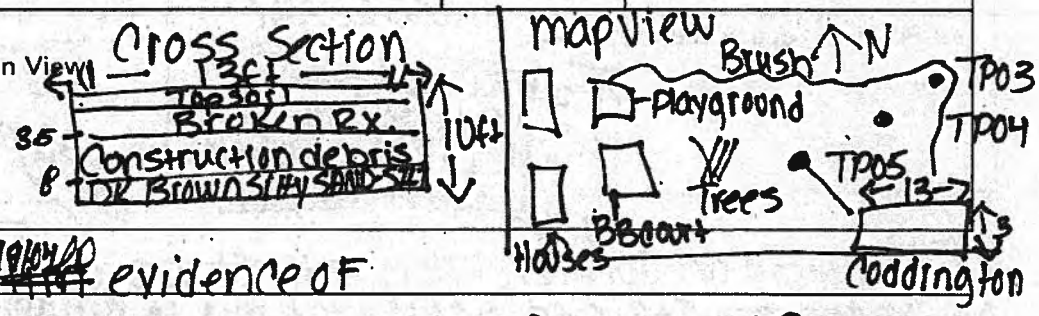
TETRA TECH NUS, INC.

TEST PIT LOG

Site Name: CCRF
Project Number: N5152-0570
Location: Newport Naval Station- Newport RI.
Test Pit No.: TP05 (~location of proposed TP02 location)
Date: 5/19/04
Field Geologist: J. Danieli

DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Topsoil	0-1 Topsoil- Brown	Fill	
		1-3.5 Gray- Broken		
2	Fill	Rock- Phyllite		
4		3.5-8.0 Brown-Gray		
6	Fill	Construction debris SAND+Gravel; Asphalt, Concrete + Rock.		
8				Native ~~Materials~~ soils-8' bgs
10	Native ~~Materials~~ soils	DK Brown - Silty SAND- SILT compact.	SM/ML	∇ ~9' bgs
		Bottom of Test Pit 10' bgs		

Test Pit Cross Section and/or Plan View



REMARKS: No visible ~~contam~~ 5/19/04 evidence of contaminated soils, No sheen or staining. Dig to GW ≅ 9.5 Ft bgs. Collect soil sample from entire pit - soil scraped from walls of pit - CCRF-S-TP05-0010 at 1150 and GW sample: CCRF-GW-TP05 at 1310 - GW silty and murky, no sheen and no odor.

PHOTO LOG:
Photo 1 to South (South wall of Pit)
Photo 2 to West (West + South wall of Pit).

TEST PIT: TP05

PAGE 1 OF 1

Tt NUS FORM 0011

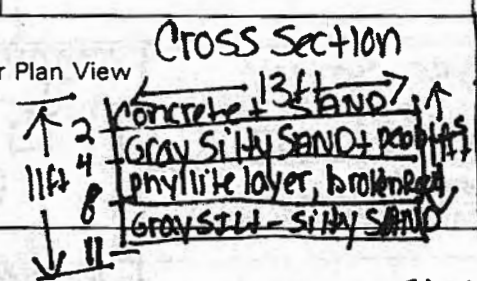
TETRA TECH NUS, INC.

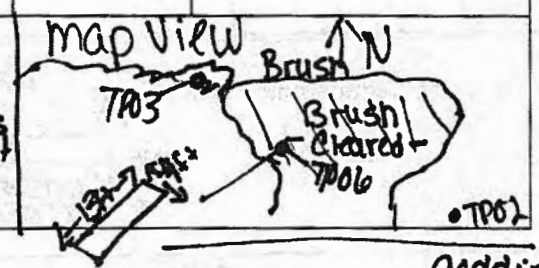
TEST PIT LOG

Site Name: CCRF
Test Pit No.: TP06
Project Number: N5152-0570
Date: 5/19/04
Location: Newport Naval Station Newport, RI.
Field Geologist: J. Danieli

DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Fill	0-2 Large Slabs of Concrete and Brown SAND w/Gravel	Fill	
2		piece of Red Rag and pieces of plastic		
4	Fill - Beach Sand	2-4 Gray-Silty Sand Round Pebbles and Stones		
6	Fill pebbles	6-6.5 - Phyllite layer - broken ROCK - Gray 6.5-8 - Gray-Green SILT		
8		w/ Rock pieces		8' ≅ Native soils
10	Native Soils.	8-11ft - Native soils smooth layer - Gray SILT -	ML/SM	V ≅ 10.5 bgs
12		Silty SAND. Bottom of Pit - 11' bgs		11'

Test Pit Cross Section and/or Plan View





REMARKS:
Large pieces on concrete - concrete slabs 0-2 ft bgs, No sheen or stained soils. Pit dug to GW = TP11 ft bgs (GW ≅ 10.5 bgs) 2 Soil samples collected - no GW. Soil samples: CCRF-S-TP06-0608 at 1439 and CCRF-S-TP06-0204 at 1509.

Codding to High Way

PHOTO LOG:
2 Photos - West Wall, 1 Photo - West + South wall and Concrete Slab, 1 photo South Wall, 1 photo North Wall, 1 photo east wall.

TEST PIT: TP06
PAGE 1 OF 1

Tt NUS FORM 0011

Headspace PID = ~~10.6~~ 1.8 ppm
FID = 10.6 ppm

TETRA TECH NUS, INC.

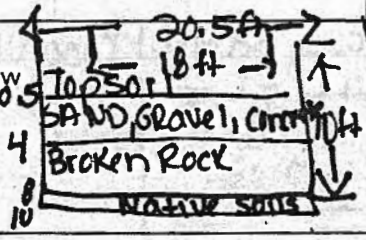
TEST PIT LOG

Site Name: CCRF | Test Pit No.: TP07
Project Number: N5152-0570 | Date: 5/20/04
Location: Newport Naval Station, Newport RI | Field Geologist: J. Danieli

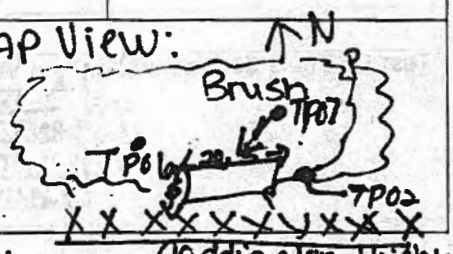
DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Topsoil	0-0.5 Topsoil - Roots	Fill	
		Loose 0.5-2.0 DK Brown		
2	Fill	SAND, GRAVEL, Rounded Stones, concrete and asphalt.		
4		2-4, same as above w/ larger pieces of Asphalt.		
6	Fill	4-6 silty SAND - pieces of Broken rock - phyllite.		
8		6-8 - Loose - same as above less Silt more pieces of broken rock.		~8' bgs - Native soils
10	Native soils	8-10 Dense - DK Brown-Gray Silty SAND - SILT	SM/ML	▽ ≅ 10' bgs
		Bottom of Test pit 10' bgs		

Test Pit Cross Section and/or Plan View

Note: deepest portion of Pit 18ft long.
Width of Pit = 5ft.



MAP View:



REMARKS: No visible evidence of contaminated soils. No sheen or staining. NO GW Sample Collected. 2 Soil Samples Collected - CCRF-S-TP07-0608 at 0810 and CCRF-S-TP07-0204 at 0840 GW-no sheen or odor, murky and silty

PHOTO LOG: 3 Photos to east, 2 Photos to North, 1 Photo into pit - to bottom, 3 Photos South, 2 Photos West

TEST PIT: TP07

PAGE 1 OF 1

Tt NUS FORM 0011

B2 = PID + FID = 0.0 ppm

Headspace: FID = 7.1 ppm
PID = 1.6 ppm

TETRA TECH NUS, INC.

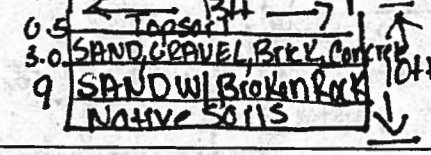
TEST PIT LOG

Site Name: CCRF
Project Number: N5152-0570
Location: Newport Naval Station - Newport RI
Test Pit No.: TP08
Date: 5/20/04
Field Geologist: J. Danieli

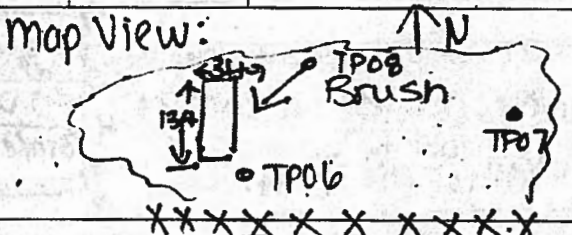
DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Topsoil	Loose. 0-0.5 – Topsoil-Roots-Brown	Fill	
		0.5-3.0 – Brown, Loose-SAND+		
2	Fill	GRAVEL w/ Brick, Asphalt+ Concrete ↓		
4		3-4 Brown Loose SAND w/ pieces of Rx - Phyllite?		
6	Fill	4-6 DK Brown – more Compacted and Silty - Broken rock - phyllite (pieces of Rock layered)		9' bgs - Native soils U ≈ 9' bgs
8				
10	Native Soils	DK-Brown Gray SILT - compact	ML	
		Bottom of Test Pit at 10' bgs		

Test Pit Cross Section and/or Plan View

Cross Section



Map View:



REMARKS: No visible contamination of soils - no sheen or staining. Collect GW Sample (depth to GW ~9' bgs) CCRF-GW-TP08 at 0930. Collect Soil sample from 2-4ft layer at 1035 - CCRF-S-TP08-0204 at 1035. Note-GW white foam on surface.

PHOTO LOG: 3 photos to East, 1 Photo North, 2 Photos South, 1 Photo West

TEST PIT: TP08

PAGE 1 OF 1

Tt NUS FORM 0011

Headspace: BZ = PID = 0.1 ppm FID = 1.1 ppm
PID = 2.0 ppm FID = 2.1 ppm

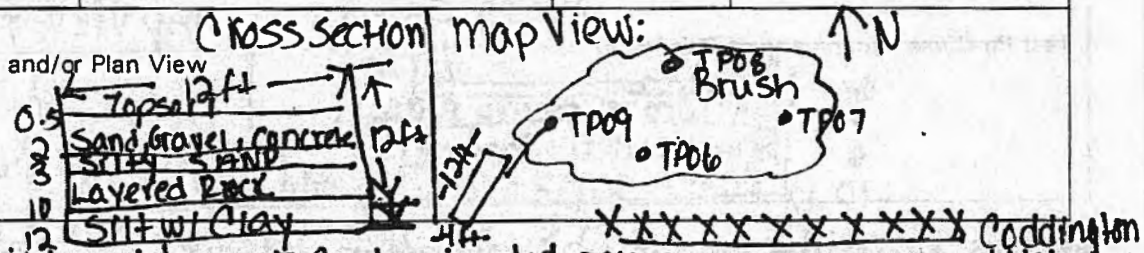
TETRA TECH NUS, INC.

TEST PIT LOG

Site Name: CCRF | Test Pit No.: TP09
Project Number: N5152-0570 | Date: 5/20/04
Location: Newport Naval Station, Newport, RI. | Field Geologist: J. Danieli

DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Topsoil	0-0.5 Topsoil/Roots	Fill	
2	Fill	0.5-2 SAND, GRAVEL, Concrete, Asphalt, Brick		
4	Fill	2-3 Brown-compact Silty SAND		
6		3-10 Pieces of Rock - Phyllite - layered - pieces of concrete and brick		
8				10ft bgs - Native soils.
10				▽ ≅ 10' bgs
12		DK Brown - DK Gray dense Silt w/ Clay - SILT	ML/MH	
		Bottom of Test Pit 12ft bgs		

Test Pit Cross Section and/or Plan View



REMARKS: No visible evidence of Contaminated soils - No sheen, no Highway Staining, NO GW sample Collected - GW silty + murky. Collect 2 soil samples. CCRF-S-TP09-1112 at 1253 and MS/MSD. Soil Material Collected from soil Pile Clumps of Gray-Clay SILT material sampled, spoon 05/20/04.

PHOTO LOG: Collect Soil Sample From 2-4ft layer CCRF-S-TP09-0204 at 1248
4 photos to North, 2 Photos to East, 3 photos to South

TEST PIT: TP09

PAGE 1 OF 1

Tt NUS FORM 0011

BZ = 4-1 FID and 0.0 PID
Headspace = PID = 0.0 + FID = 182

Note: Samples collected From Silt/Clay 11-12' bgs - request by Patti RIDEM.

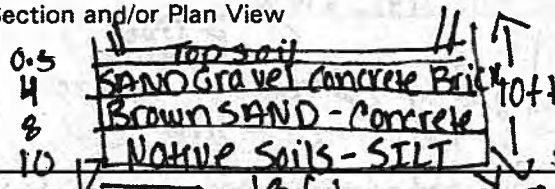
TETRA TECH NUS, INC. — **TEST PIT LOG**

Site Name: CCRF — Test Pit No.: TP10
Project Number: 5152-0570 — Date: 5/20/04
Location: Newport Naval Station Newport RI — Field Geologist: J. Danieli

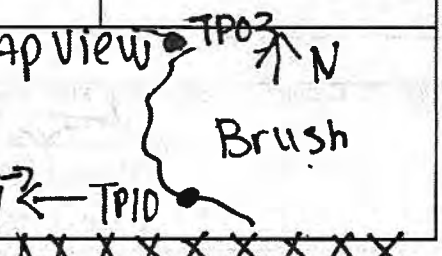
DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Topsoil	0-0.5' Top Soil/Roots	Fill	
2	Fill	0.5-4' Brown, loose, SAND, GRAVEL, Concrete, Brick - DK Brown layer of Soil - Collect Sample ~4'.		
4				
6		4-8' Brown SAND - pieces of concrete		
8				
10	Native Soils	DK Brown - Gray -Silty SAND - SILT	SM/ML	8' bgs - Native ~~materials~~ Soils
12		Bottom of Test Pit at 10 feet bgs.		V ≅ 10' bgs

Test Pit Cross Section and/or Plan View

Cross Section:



MAP View



REMARKS: No visible evidence of contaminated soils. Coddington Cove NO GW Sample Collected GW at ~10' bgs. - sides of pit falling in filling in GW at bottom of Pit. 2 Soil samples collected - CCRF-S-TP10-0204 at 1450 and CCRF-S-TP10-0406 at 1525. DP02 From CCRF-S-TP10 at 1505.

PHOTO LOG: 2 Photos to South, 2 photos to West, 3 photos to North, and 1 photo to East. TEST PIT: TP10 PAGE 1 OF 1

Tt NUS FORM 0011

BZ = FID = 0.2 + PID = 0.0
Headspace - PID = 0.0 ppm and FID = 0.2 ppm.

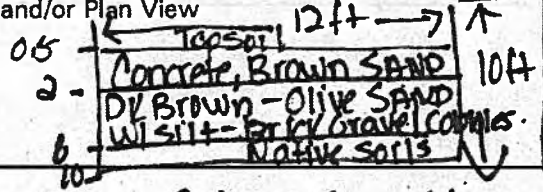
TETRA TECH NUS, INC. — **TEST PIT LOG**

Site Name: CCRF — Test Pit No.: TP11
Project Number: N5152-0570 — Date: 5/20/04
Location: Newport Naval Station Newport RI — Field Geologist: J. Danieli

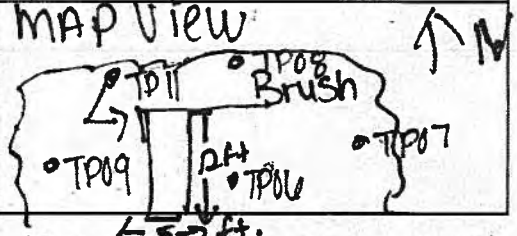
DEPTH (feet)	LITHOLOGY CHANGE (Depth, feet)	MATERIAL DESCRIPTION (Soil Density/Consistency, Color)	USCS	REMARKS
0	Topsoil / Fill	0-0.5 - Topsoil / Roots	Fill	
2	↓	0.5-2.0 large slabs of Concrete, w/ Brown SAND w/ smaller pieces of concrete, Gravel + Brick + Asphalt.		
4	Fill	2-8' DK Brown-Olive SAND w/ silt-Gravels, cobbles, brick, pieces of concrete		
6	↓	↓	↓	
8	↓	↓	↓	8' bgs - Native Soils
10	Native Soils	DK Brown Silty SAND	SM	TD ≅ 10' bgs
		Bottom of Pit at 10ft bgs.		

Test Pit Cross Section and/or Plan View

Cross Section



MAP View



REMARKS: No SAMPLES Collected. - No visible evidence of contaminated soils - no sheen, no staining.
(Additional Pit Suggested by Paul Culpa - RI DEM)

PHOTO LOG: 2 Photos to North, 3 photos to East, 3 photos to South, 2 photos to West.

TEST PIT: TP11

PAGE 1 OF 1

Tt NUS FORM 0011

Solid Phase
Log Sheets

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP02-0001

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: N/A (if applicable)

Sample Method: Test Pit - Scoop w/ excavator bucket
Depth Sampled: ~~0-1~~ 0-1 feet below ground surface
Sample Date & Time: 5/18/04 1410 hours Dup NA hours
Sampler(s): J. Daniels

Data Recorded By: J. Daniels (Signature)

PID/OVA Monitor Reading: FID=0.0 ppm
PID=0.0 ppm

TYPE OF SAMPLE: (Check all that apply)

X Soil	___ Trip Blank*
___ Sediment	___ Rinsate Blank*
___ Lagoon/Pond	___ Field Duplicate collected
X Grab	___ Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See TP02 - Test Pit log For details

SAMPLE DATA/REMARKS: Sample collect from top 1 foot of ~~test~~ pit - from area where asphalt, brick and ash present - sample scooped by excavator bucket and grabbed from bucket to fill sample bottles.

ANALYSIS	BOTTLE LOT NO.
VOC	
GRO	
Total metals	
SVOCS	
Pesticides	
PCB	
TPH	
% moisture	

NOTES/SKETCH: see test pit log: TP02

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF-S-TP03-0010
Sample ID: CCRF

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: DP01 (if applicable)

Sample Method: Test Pit - Scoop w/ excavator bucket
Depth Sampled: 0-10 feet below ground surface
Sample Date & Time: 5/19/09 0830 hours Dup 0835 hours
Sampler(s): J. Daniell

Data Recorded By: J. Daniell (Signature)

PID/OVA Monitor Reading: FID: 112.8 ppm
PID: 6.1 ppm

TYPE OF SAMPLE: (Check all that apply)

- X Soil
- ___ Sediment
- ___ Lagoon/Pond
- X Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- X Field Duplicate collected
- ___ Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See Test Pit Log TP03 for details

SAMPLE DATA/REMARKS: Sample collected from test pit walls - scooped by excavator bucket and then grabbed from bucket to fill sample jars. GW at ~ 10 ft bgs

ANALYSIS	BOTTLE LOT NO.
VOCs	
GRO	
Total Metals	
SVOCs	
Pesticides	
PCB	
TPH	
% moisture	

NOTES/SKETCH: See Test Pit 3 Log - TP03

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP05-0010

Tetra Tech NUS Charge No.: N5152-0570
QC Information: NA (if applicable)

Sample Method: Test Pit Scoop w/ excavator bucket
Depth Sampled: 0-10 - feet
Sample Date & Time: 5/19/04 1150 hours Dup NA hours
Sampler(s): J. Daniels

Data Recorded By: J. Daniels
Signature

PID/OVA Monitor Reading: FID= 153 ppm
PID= 18.1 ppm

TYPE OF SAMPLE: (Check all that apply)

- X Soil
- ___ Sediment
- ___ Lagoon/Pond
- ___ Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate collected
- ___ Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See Test Pit log TP05 for details

SAMPLE DATA/REMARKS: Samples collected from test pit walls - scooped by excavator bucket and then grabbed from center of bucket to fill sample bottles, GW ~ 10' bgs

ANALYSIS	BOTTLE LOT NO.
VOCs	
SVOCs	
GRO	
Pesticides	
PCBs	
TPH	
Total Metals	
% Moisture	

NOTES/SKETCH: see TP05 Test Pit log for details

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP04-0010

Tetra Tech NUS Charge No.: N5152-0570
QC Information: NA (if applicable)

Sample Method: Test Pit Scoop w/ excavator bucket
Depth Sampled: 0-10 - feet below ground surface
Sample Date & Time: 5/19/04 1030 hours Dup NA hours
Sampler(s): J. Daniell

Data Recorded By: J. Daniell
Signature

PID/OVA Monitor Reading: FID=172.0 ppm
PID=0.0 ppm

TYPE OF SAMPLE: (Check all that apply)

- X Soil
- ___ Sediment
- ___ Lagoon/Pond
- X Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate collected
- ___ Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See Test Pit log TP04 for details

SAMPLE DATA/REMARKS: Sample collected from test pit walls - scooped by excavators bucket and then grabbed from bucket to fill sample bottles BW @ 10 ft-bgs

ANALYSIS	BOTTLE LOT NO.
VOCs	
SVOCs	
GRO	
Pesticides	
PCBs	
TPH	
TAL metals	
% moisture	

NOTES/SKETCH: see TP04-Test Pit log for details.

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP06-0204

Tetra Tech NUS Charge No.: N5152-0570
QC Information: NA (if applicable)

Sample Method: Scoop W/ 8oz Glass jar attached to plastic
Depth Sampled: 2-4 feet below ground surface PVC pole
Sample Date & Time: 5/19/04 1509 hours Dup NA hours
Sampler(s): J. Daniell

Data Recorded By: J Daniell
Signature

PID/OVA Monitor Reading: FID= 10.6 ppm } Headspace For 0-10' OF soil From Pit
PID= 1.8 ppm }

TYPE OF SAMPLE: (Check all that apply)

- X Soil
- ___ Sediment
- X Lagoon/Pond
- X Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate collected
- ___ Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See TP06 - Test Pit log For details.

SAMPLE DATA/REMARKS: Sample Collected - 2-4 ft Fill layer. OF Gray-silty SAND W/ Beach Stones ~~SAND W/ Gravel - Pieces of Rag~~ and pieces of plastic @ 5/19/04 - Sample scooped from layer W/ glass 8oz amber jar attached to PVC pole.

ANALYSIS	BOTTLE LOT NO.
VOCs	
SVOCs	
GRO	
Pesticides	
PCBs	
TPH	
Total metals	
% Moisture	

NOTES/SKETCH: See TP06 - Test pit log for details

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP06-0008

Tetra Tech NUS Charge No.: N5152-0570
QC Information: NA (if applicable)

Sample Method: Scoop w/ 8oz Glass jar attached to plastic PVC pole
Depth Sampled: 0-8 feet below ground surface
Sample Date & Time: 5/19/04 1439 hours Dup NA hours
Sampler(s): J. Daniell

Data Recorded By: J. Daniell
Signature

PID/OVA Monitor Reading: FID = 10.6 ppm } headspace
PID = 1.8 ppm } for 0-10' of soil from pit.

TYPE OF SAMPLE: (Check all that apply)

- X Soil
- ___ Sediment
- ___ Lagoon/Pond
- X Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate collected
- ___ Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See TP06- Test pit log

SAMPLE DATA/REMARKS: Sample collected from 6-8ft fill layer of Gray-Green Silt w/ broken pieces of rock-phyllite - sample scooped from layer w/ 8oz glass amber jar attached to plastic PVC pole.

ANALYSIS	BOTTLE LOT NO.
VOCs	
SVOCs	
GRO	
Pesticides	
PCBs	
TPH	
Total metals	
% moisture	

NOTES/SKETCH: See TP06 - Test Pit log for details

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP07-0204

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: NA (if applicable)

Sample Method: Scoop w/ 8oz jar attached to PVC pole From 2-4' layer along Pit wall.
Depth Sampled: 2-4 feet below ground surface.
Sample Date & Time: 5/20/04 0848 hours Dup NA hours
Sampler(s): J. Danieli + G. Glennon

Data Recorded By: J. Danieli
Signature

PID/OVA Monitor Reading: FID= 7.1 ppm } Headspace From Soils 0-10' bgs from entire Pit.
PID= 1.6

TYPE OF SAMPLE: (Check all that apply)

X	Soil		Trip Blank*
	Sediment		Rinsate Blank*
	Lagoon/Pond		Field Duplicate collected
X	Grab		Other (Specify):

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See Test Pit log TP07

SAMPLE DATA/REMARKS: Sample Collected from 2-4' fill layer of SAND w/ Gravel, concrete, and Asphalt - Sample scooped from 2-4 ft layer w/ glass 8oz Amber jar attached to PVC pole.

ANALYSIS	BOTTLE LOT NO.
VOCs	
SVOCs	
GRO	
Pesticides	
PCBs	
TPH	
Total metals	
% moisture	

NOTES/SKETCH: See TP07 - Test Pit log TP07.

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP07-0608

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: NA (if applicable)

Sample Method: Scoop w/ 8oz jar attached to PVC pole-from
Depth Sampled: 6-8 feet below g.s. 6-8' layer along pit wall
Sample Date & Time: 5/24/04 0810 hours Dup NA hours
Sampler(s): J. Daniel / G. Glennon

Data Recorded By: J. Daniell
Signature

PID/OVA Monitor Reading: PID = 1.6 ppm
FID = 7.1

} Headspace From soils 0-10ft bgs from entire pit.

TYPE OF SAMPLE: (Check all that apply)

X	Soil	___	Trip Blank*
___	Sediment	___	Rinsate Blank*
___	Lagoon/Pond	___	Field Duplicate collected
X	Grab	___	Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See Test Pit log TP07

SAMPLE DATA/REMARKS: Sample collected from 6-8' fill layer of - silty SAND - pieces of Broken rock ~~and~~ phyllite? - sample scooped from layer w/ glass-8oz amber jar attached to PVC pole.

ANALYSIS	BOTTLE LOT NO.
VOCs	
SVOCs	
GRO	
Pesticides	
PCBs	
TPH	
Total Metals	
% moisture	

NOTES/SKETCH: see TP07 - Test Pit Log for ~~TP07~~ details

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP08-0204

Tetra Tech, NUS Job No./PMS
QC Information: NA (if applicable)

Sample Method: Scoop w/8oz jar attached to PVC pole from wall of Test Pit at 2-4ft layer
Depth Sampled: 2-4 feet bgs
Sample Date & Time: 5/20/04 1035 hours Dup NA hours
Sampler(s): J. Danieli / G Glennon

Data Recorded By: J. Danieli
Signature

PID/OVA Monitor Reading: PID = 20 ppm } Headspace of soils 0-10ft from Pit
FID = 2.1 ppm }

TYPE OF SAMPLE: (Check all that apply)

X	Soil		Trip Blank*
	Sediment		Rinsate Blank*
	Lagoon/Pond		Field Duplicate collected
X	Grab		Other (Specify):

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See Test Pit log - TP08

SAMPLE DATA/REMARKS: Sample Collected from 2-4' fill layer of SAND, GRAVEL, Asphalt, brick and Concrete, sample scooped from layer with glass-8oz amber jar attached to PVC Pole.

ANALYSIS	BOTTLE LOT NO.
VOC's	
SVOCS	
GRO	
Pesticides	
PCBs	
TPH	
Total Metals	
% Moisture	

NOTES/SKETCH: See TP08 - Test Pit log

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP09-0204

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: NA (if applicable)

Sample Method: Scoop w/ 8oz jar attached to PVC Pole
Depth Sampled: 2-4 feet below ground surface.
Sample Date & Time: 5/20/04 1248 hours Dup NA hours
Sampler(s): J. Danieli

Data Recorded By: J. Danieli (Signature)

PID/OVA Monitor Reading: PID = 0.0 ppm, FID = 182 ppm } Headspace of soils from 0-10 of Pit

TYPE OF SAMPLE: (Check all that apply)

- [X] Soil
- [ ] Sediment
- [ ] Lagoon/Pond
- [X] Grab
- [ ] Trip Blank*
- [ ] Rinsate Blank*
- [ ] Field Duplicate collected
- [ ] Other (Specify):

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See Test Pit log - TP09

SAMPLE DATA/REMARKS: Sample collected from 2-4ft Fill layer of SAND, GRAVEL, Asphalt, Brick, and Rock layer (Phyllite layer). w/ 8oz amber glass jar attached to PVC pole. ↑ Broken/Crushed

ANALYSIS	BOTTLE LOT NO.
VOCs	
GRO	
SVOCs	
Pesticides	
PCB	
TPH	
Total metals	
% moisture	

NOTES/SKETCH: See TP09 - Test Pit log for details.

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP09-1112

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: MS/MSD (if applicable)

Sample Method: Soils scooped by Excavator bucket - Grab From Soil Pile
Depth Sampled: 11-12 feet below ground surface
Sample Date & Time: 5/20/04 1253 hours Dup NA hours
Sampler(s): J. Daniels

Data Recorded By: J. Daniels (Signature)

PID/OVA Monitor Reading: FID = 182.0 ppm, PID = 0.0 ppm } Headspace From 0-12" of soils

TYPE OF SAMPLE: (Check all that apply)

- X Soil
- ___ Sediment
- ___ Lagoon/Pond
- X Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate collected
- ___ Other (Specify): MS/MSD

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See TP09-Test Pit log

SAMPLE DATA/REMARKS: Sample Silt/Clay clumps - on top of soil pile - pulled out of pit (11-12' bgs) by excavator.

ANALYSIS	BOTTLE LOT NO.
VOC's	
SVOCs	
Pesticides	
PCBs	
TPH	
GRO	
Total metals	
% moisture	

NOTES/SKETCH: See TP09-Test Pit Log

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF
Sample ID: CCRF-S-TP10-0204

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: _____ (if applicable)

Sample Method: Scoop
Depth Sampled: 2-4 feet below ground surface
Sample Date & Time: 5/20/04 1525 hours Dup N/A hours
Sampler(s): J. Danieli + G. Glennon

Data Recorded By: J. Danieli
Signature

PID/OVA Monitor Reading: FID = 0.2 ppm } Headspaces of soils 0-10' from pit
PID = 0.0 }

TYPE OF SAMPLE: (Check all that apply)

- X Soil
- ___ Sediment
- ___ Lagoon/Pond
- X Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate collected
- ___ Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See TP10 - Test Pit log

SAMPLE DATA/REMARKS: Sample collected from 2-4 ft fill layer of DK Brown SAND layer w/ Concrete, Brick, Sample scooped from layer with 8oz glass amber jar attached to PVC pole.

ANALYSIS	BOTTLE LOT NO.
VOCs	
GRO	
SVOCs	
Pesticides	
PCBs	
TPH	
Total metals	
% moisture	

NOTES/SKETCH: See TP10 - test Pit log

Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: CCRF-S. TP10-0406F-CCRF
Sample ID:

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: DP02 (if applicable)

Sample Method: Scoop w/ 8oz jar-attached to PVC Pole
Depth Sampled: 4-6 feet below ground surface. From wall of Test Pit 4-6 layer.
Sample Date & Time: 5/20/04 1450 hours Dup 1505 hours
Sampler(s): J. Danieli + G. Glennon

Data Recorded By: J. Danieli (Signature)

PID/OVA Monitor Reading: FID= 0.2 ppm, PID= 0 } Headspace From 0-10' OF SOILS

TYPE OF SAMPLE: (Check all that apply)

- X Soil
- ___ Sediment
- ___ Lagoon/Pond
- X Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- X Field Duplicate collected
- ___ Other (Specify):

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) See TP10 - Test Pit log

SAMPLE DATA/REMARKS: Sample collected from 4-6 feet fill layer OF - Brown SAND + pieces OF concrete, sample scooped from layer with an 8oz glass amber jar attached to PVC pole.

ANALYSIS	BOTTLE LOT NO.
VOCs	
GRO	
SVOCs	
Pest.	
PCBs	
TPH	
Total metals	
% moisture	

NOTES/SKETCH: See TP10 Test Pit log for details

Tt NUS Form 0005A

Liquid Phase
Log Sheets

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - LIQUID PHASE

Site Name: CCRF
Sample ID: CCRF-GW-TP01

Tetra Tech NUS Charge No. N5152-0570
QC Information: NA (if applicable)

Sample Method/Device: Direct dip From Test Pit
Depth Sampled: ~8 feet Total Depth ~8 feet (SW Only)
Sample Date & Time: 5/18/04 1254 hours
Sampler(s): J. Danieli

Recorded By: J. Danieli
Signature

TYPE OF SAMPLE: (Check all that apply)

- [X] Groundwater
- [ ] Surface Water
- [X] Residential Supply
- [X] Grab
- [ ] Composite
- [ ] Trip Blank*
- [ ] Rinsate Blank*
- [ ] Field Duplicate Collected
- [ ] Other (Specify):

* include sample source & lot No.

WELL PURGE DATA: NA

Micro Tip/OVA Monitor Reading: 0 ppm

Well Depth	feet	Purge Start	hrs
Inside Diameter	inches	Purge Stop Time	hrs
		NA	
Water Level	feet	Total Gallons Purged	
Well Volume	gal.	Purge Method	NA

Color: _____ Turbidity: CLR/SL CLDY/CLDY/OPAQ

Sampling/Purge Data:

Vol. #	Temp °C	pH	Spec. Cond.	DO
0				
1				
3				
4		NA	NA	

ANALYSIS	BOTTLE LOT NO.	TRAFFIC REPORT NO.		COMMENTS
		ORGANIC	INORGANIC	
VOA				Collect Sample ~2hrs after
ORO				Soil Sample allow For water
SVOC				to collect in Pit.
Pest				Note: Slight Sheen on water in hole-
PCB				water Silty & murky no odor
TPH				
Total Metals				

TtNUS Form 0004

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - LIQUID PHASE

Site Name: CCRA
Sample ID: CCRA-GW-TP02

Tetra Tech NUS Job No./PMS: N5152-0570
QC Information: NA. (if applicable)

Sample Method/Device: Direct dip From Test Pit
Depth Sampled: ~10 feet bgs Total Depth ~10 feet (SW Only)
Sample Date & Time: 5/18/04 1515 hours
Sampler(s):

Date Recorded By: J. Danieli Signature: J. Danieli

bgs = below Ground Surface

TYPE OF SAMPLE: (Check all that apply)

- X Groundwater
- ___ Surface Water
- ___ Residential Supply
- X Grab
- ___ Composite
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate Collected
- ___ Other (Specify):

*include sample source & lot No.

WELL PURGE DATA: NA.

Well Depth	feet	Purge Start	hrs
Inside Diameter	inches	Purge Stop Time	hrs
		1015 5/18/04	
Water Level	feet	Total Gallons Purged	
Well Volume	gal.	Purge Method	

Color: _____ Turbidity: CLR/SL CLDY/CLDY/OPAQ

Micro Tip/OVA Monitor Reading: ppm

Sampling/Purge Data: NA

Vol. #	Temp °C	pH	Spec. Cond.	DO
0				
1				
3				
4				

NA 5/18/04

ANALYSIS	BOTTLE LOT NO.	TRAFFIC REPORT NO.		COMMENTS
		ORGANIC	INORGANIC	
VOC				Collect sample ~1hr after
GRO				soil sample at TP02.
SVOC				Water silty, turbid, no sheen
Pest				and no odor.
PCB				
TPH				
Total Metals				

Tt NUS Form 0004

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - LIQUID PHASE

Site Name: CCRF
Sample ID: CCRF-GW-TP03

Tetra Tech NUS Charge No. N5152-0570
QC Information: NA (if applicable)

Sample Method/Device: Direct Dip From Test Pit
Depth Sampled: ~10 feet bgs Total Depth ~10 feet (~~SW Only~~) bgs
Sample Date & Time: 5/19/04 0930 hours
Sampler(s): J. Daniels

Recorded By: _____ Signature

bgs = below ground surface

TYPE OF SAMPLE: (Check all that apply)

- X Groundwater
- ___ Surface Water
- ___ Residential Supply
- X Grab
- ___ Composite
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate Collected
- ___ Other (Specify):

* Include sample source & lot No.

WELL PURGE DATA: NA

Micro Tip/OVA Monitor Reading: NA ppm

Well Depth	feet	Purge Start	hrs
Inside Diameter	Inches	Purge Stop Time	hrs
Water Level	feet	Total Gallons Purged	
Well Volume	gal.	Purge Method	

Color: _____ Turbidity: CLR/SL CLDY/CLDY/OPAQ

Sampling/Purge Data:

Vol. #	Temp °C	pH	Spec. Cond.	DO
0				
1				
3				
4				

ND 5/19/04

ANALYSIS	BOTTLE LOT NO.	TRAFFIC REPORT NO.		COMMENTS
		ORGANIC	INORGANIC	
VOC				Collect sample ~1hr after soil sample at TP03 Water Silty, turbid, no sheen and no odor.
SVOC				
CLO Pesticide				
PCB				
TPH				
Total Metals				

TtNUS Form 0004

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - LIQUID PHASE

Site Name: CCRF
Sample ID: CCRF-GW-TP04

Tetra Tech NUS Job No./PMS: N5152-0570
QC Information: DP01 (if applicable)

Sample Method/Device: Direct dip from Test Pit
Depth Sampled: 10 feet bgs Total Depth 10 feet ~~(SW Only) bgs~~
Sample Date & Time: / 1120 hours
Sampler(s): J. Daniell

Date Recorded By: J. Daniell
Signature

bgs = below ground surface DP01 @ 1120

TYPE OF SAMPLE: (Check all that apply)

- X Groundwater
- ___ Surface Water
- ___ Residential Supply
- X Grab
- ___ Composite
- ___ Trip Blank*
- ___ Rinsate Blank*
- A Field Duplicate Collected
- ___ Other (Specify):

*include sample source & lot No.

WELL PURGE DATA: NA

Micro Tip/OVA Monitor Reading: NA ppm

Well Depth	feet	Purge Start	hrs
Inside Diameter	Inches	Purge Stop Time	hrs
Water Level	feet	Total Gallons Purged	10/5/19104
Well Volume	gal.	Purge Method	

Color: _____ Turbidity: CLR/SL CLDY/CLDY/OPAQ

Sampling/Purge Data:

Vol. #	Temp °C	pH	Spec. Cond.	DO
0				
1				
3				
4				

10/5/19104

ANALYSIS	BOTTLE LOT NO.	TRAFFIC REPORT NO. ORGANIC	TRAFFIC REPORT NO. INORGANIC	COMMENTS
VOC				Collect sample ~ 40 min
GRO				after soil sample.
SVOCs				
Pes				
PCB				
Total metals				

Tt NUS Form 0004

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - LIQUID PHASE

Site Name: CCRF
Sample ID: CCRF-GW-TP05

Tetra Tech NUS Job No./PMS: N5152-0570
QC Information: NA (if applicable)

Sample Method/Device: Direct drip From Test pit
Depth Sampled: 10 feet bgs Total Depth ~10 feet (SW Only) bgs
Sample Date & Time: 5/19/04 1310 hours
Sampler(s): J. Danieli + S. Parker

Date Recorded By: J. Danieli (Signature)

TYPE OF SAMPLE: (Check all that apply)

- X Groundwater
- ___ Surface Water
- ___ Residential Supply
- ___ Grab
- ___ Composite
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate Collected
- ___ Other (Specify):

*include sample source & lot No.

WELL PURGE DATA: NA

Well Depth	feet	Purge Start	hrs
Inside Diameter	Inches	Purge Stop Time	hrs
Water Level	feet	Total Gallons Purged	10/5/19/04
Well Volume	gal.	Purge Method	

Color: _____ Turbidity: CLR/SL CLDY/CLDY/OPAQ

Micro Tip/OVA Monitor Reading: _____ ppm

Sampling/Purge Data: NA

Vol. #	Temp °C	pH	Spec. Cond.	DO
0				
1				
3				
4				

10/5/19/04

ANALYSIS	BOTTLE LOT NO.	TRAFFIC REPORT NO. ORGANIC	TRAFFIC REPORT NO. INORGANIC	COMMENTS
VOC				Collect GW sample ~1hr 40min after soil sample from test pit. GW - silty/murky, no sheen and no odor.
GRO/DRO				
SVOCs				
Pesticides				
PCB				
TPH				

Tt NUS Form 0004

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - LIQUID PHASE

Site Name: CCRF
Sample ID: CCRF-GW-TB08
Tetra Tech NUS Job No./PMS: N5152-0570
QC Information: MS/MSD (if applicable)

Sample Method/Device: direct dip into Test Pit
Depth Sampled: 9 feet bgs Total Depth 9 feet (~~SW Only~~) bgs
Sample Date & Time: 5/20/04 hours
Sampler(s): J. Danieli & G. Glennon.
Date Recorded By: J Danieli / Danieli
Signature

bgs= below Ground surface.

TYPE OF SAMPLE: (Check all that apply)

- X Groundwater
- ___ Surface Water
- ___ Residential Supply
- ___ Grab
- ___ Composite
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate Collected
- ___ Other (Specify):

*include sample source & lot No.

WELL PURGE DATA: NA

Micro Tip/OVA Monitor Reading: NA ppm

Well Depth	feet	Purge Start	hrs
Inside Diameter	Inches	Purge Stop Time	hrs
		10 5/20/04	
Water Level	feet	Total Gallons Purged	
Well Volume	gal.	Purge Method	

Color: _____ Turbidity: CLR/SL CLDY/CLDY/OPAQ

Sampling/Purge Data: NA

Vol. #	Temp °C	pH	Spec. Cond.	DO
0				
1				
3				
4				

10 5/20/04

ANALYSIS	BOTTLE LOT NO.	TRAFFIC REPORT NO.		COMMENTS
		ORGANIC	INORGANIC	
VOCs				
GRO				
SVOCs				
Pest				
PCB				
TPH				
Total Metals.				

Tt NUS Form 0004

3. Sample Log Sheets (Surface Soils)

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: Coddington Cove Rubble Fill Area
Sample ID: CCRF-JS-SS01-0001

Tetra Tech NUS Charge No. N5152-0570
QC Information: NA (if applicable)

Sample Method: Stainless Steel Hand Auger
Depth Sampled: 0-1 feet below ground surface
Sample Date & Time: 7/28/04 1004 hours Dup NA hours
Sampler(s): J. Danieli, K. O'Neill

Data Recorded By: Jessica Danieli
Signature

PID/OVA Monitor Reading: NA ppm

TYPE OF SAMPLE: (Check all that apply)

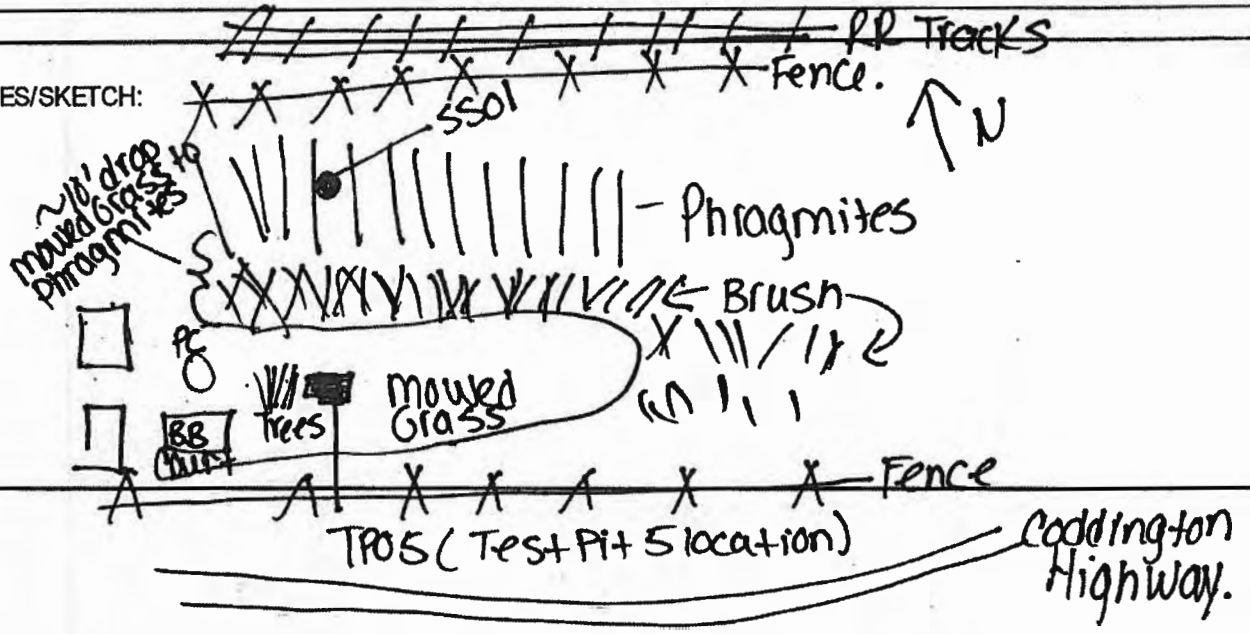
- X Soil
- ___ Sediment
- ___ Lagoon/Pond
- ___ Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate collected
- ___ Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) Wet, Dk Brown, Sandy Silt, w/ roots, plant fibers, roots, - mucky material

SAMPLE DATA/REMARKS: GW fills in hole - standing water in hole after 20 minutes- ~2ft from surface.

ANALYSIS	BOTTLE LOT NO.
VOC	
GRO	
DRO	
TPH	
metals	
SVOCs	
pesticides	
PCB	

NOTES/SKETCH:



Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: Coddington Cove Rubble Fill Area Tetra Tech NUS Charge No. N5152-0570
Sample ID: CCRF-S-SS02-0001 QC Information: DUPOT (if applicable)

Sample Method: Hand Auger.
Depth Sampled: 0-1 feet below ground surface
Sample Date & Time: 7/28/04 1050 hours Dup 1110 hours
Sampler(s): J. Danieli, K O'Neill

Data Recorded By: Jessica Danieli (Signature)

PID/OVA Monitor Reading: NA ppm

TYPE OF SAMPLE: (Check all that apply)

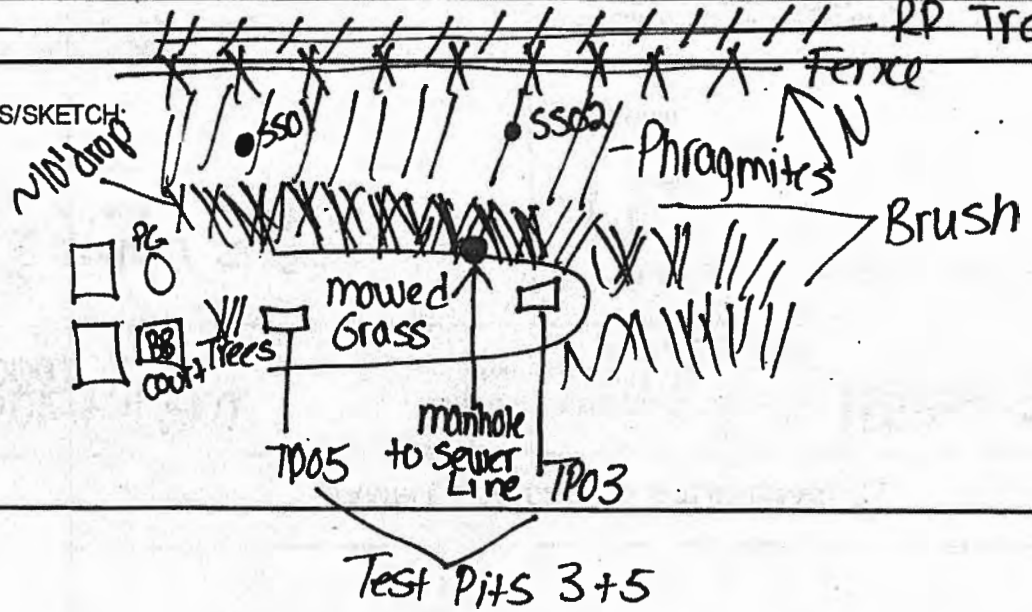
- [X] Soil
- [ ] Sediment
- [ ] Lagoon/Pond
- [ ] Grab
- [ ] Trip Blank*
- [ ] Rinsate Blank*
- [X] Field Duplicate collected
- [ ] Other (Specify):

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) Wet SILT w/ trace sand and Clay, Brown, plant Fibers, roots.

SAMPLE DATA/REMARKS: Standing water in hole.

ANALYSIS	BOTTLE LOT NO.
VOC	
SVOC	
DRO	
TPH	
metals	
SVOCs	
Pesticides	
PCB	

NOTES/SKETCH:



Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: Coddington Cove Rubble Fill Area
Sample ID: CCRF-S-SS03-0001

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: NA (if applicable)

Sample Method: Hand Auger
Depth Sampled: 0-1 feet below ground surface
Sample Date & Time: 7/28/04 1205 hours Dup NA hours
Sampler(s): J. Daniell, K. O'Neill

Data Recorded By: J. Daniell (Signature)

PID/OVA Monitor Reading: NR ppm

TYPE OF SAMPLE: (Check all that apply)

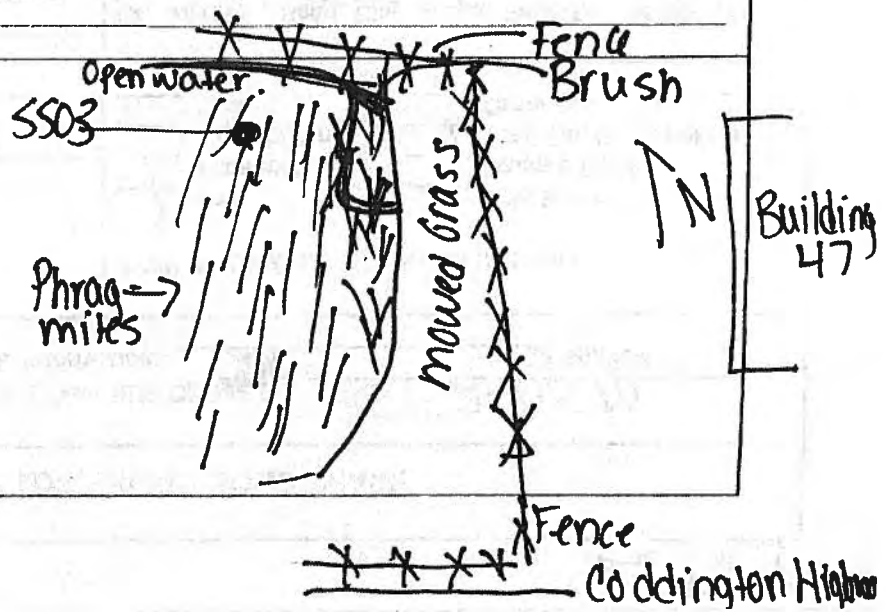
- [Y] Soil
- [ ] Sediment
- [ ] Lagoon/Pond
- [ ] Grab
- [ ] Trip Blank*
- [ ] Rinsate Blank*
- [ ] Field Duplicate collected
- [ ] Other (Specify):

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) moist, DK. Brown, Silty, with roots + plant fibers

SAMPLE DATA/REMARKS: No Standing Water in hole

ANALYSIS	BOTTLE LOT NO.
VOC	
GRO	
DRO	
TPH	
metals	
SVOCs	
Pesticides	
PCB	

NOTES/SKETCH:



Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: Coddington Cove Rubble Fill Area
Sample ID: CCRF-SD-SS04-0001
Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: MS/MSD (if applicable)

Sample Method: Hand Auger
Depth Sampled: 0-1 feet below ground surface
Sample Date & Time: 7/28/04 ~~1235~~ hours 1235 Dup NA hours
Sampler(s): J. Danieli, K. O'Neill

Data Recorded By: Jessica Danieli (Signature)

PID/OVA Monitor Reading: NR ppm

TYPE OF SAMPLE: (Check all that apply)

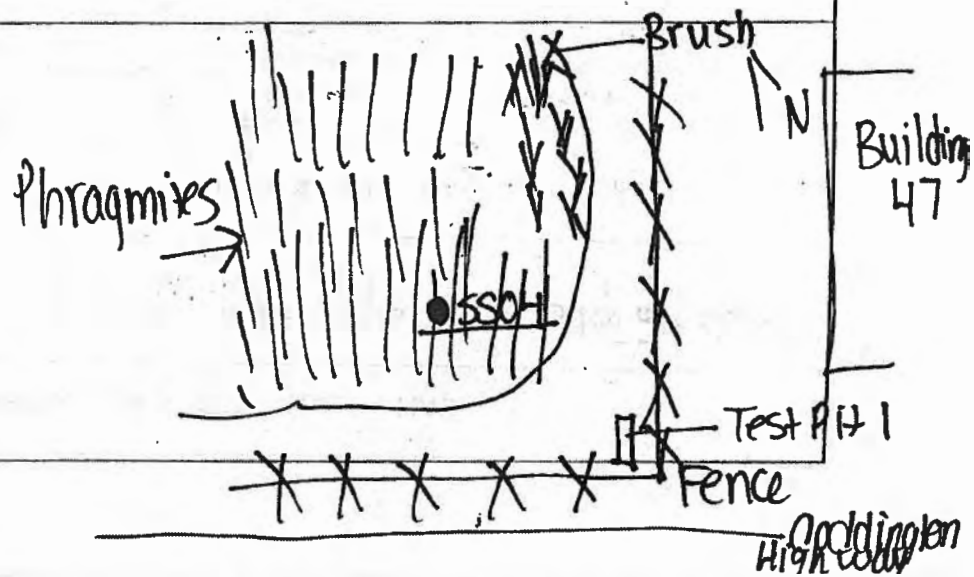
- X Soil
- ___ Sediment
- ___ Lagoon/Pond
- ___ Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate collected
- ___ Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) Wet, Dark Brown, sandy SILT, twigs, roots and plant fibers.

SAMPLE DATA/REMARKS: Standing water in Augered hole.

ANALYSIS	BOTTLE LOT NO.
VOC	
GRO	
DRO	
TPH	
metals	
SVOCs	
Pesticides	
PCB	

NOTES/SKETCH:



Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: Caddington Cove Rubble Fill Area
Sample ID: CCRF-S-5505-0001

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: NA (if applicable)

Sample Method: Hand Auger
Depth Sampled: 0-1 feet below ground surface
Sample Date & Time: 7/28/04 1351 hours Dup NA hours
Sampler(s): J. Daniell, K ONeill

Data Recorded By: Jessica Daniell (Signature)

PID/OVA Monitor Reading: NA ppm

TYPE OF SAMPLE: (Check all that apply)

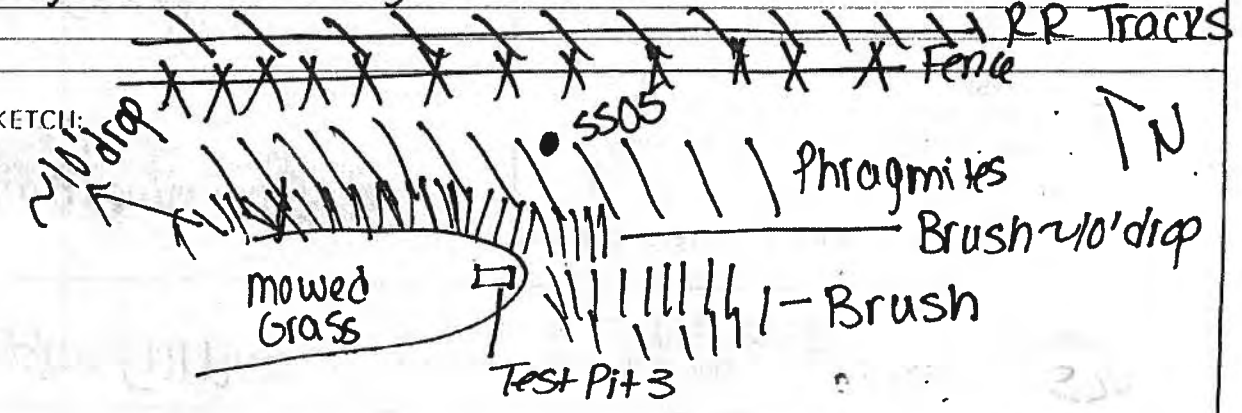
- X Soil
- ___ Sediment
- ___ Lagoon/Pond
- ___ Grab
- ___ Trip Blank*
- ___ Rinsate Blank*
- ___ Field Duplicate collected
- ___ Other (Specify): ___

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) Brown, clayey-silt, roots, plant fibers, wet

SAMPLE DATA/REMARKS: Standing water in Augered hole.

ANALYSIS	BOTTLE LOT NO.
VOC	
GRO	
BRO	
TPH	
metals	
SVOCs	
Pesticides	
PCBs	

NOTES/SKETCH:



Tt NUS Form 0005A

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: Coddington Cove Rubble Fill Area
Sample ID: CCRF-SS-SS06-0001

Tetra Tech, NUS Job No./PMS: N5152-0570
QC Information: NA (if applicable)

Sample Method: Hand Auger
Depth Sampled: 0-1 feet below ground surface
Sample Date & Time: 7/28/04 1420 hours Dup NA hours
Sampler(s): J. Danieli, K. ONeill

Data Recorded By: Jessica Danieli (Signature)

PID/OVA Monitor Reading: NA ppm

TYPE OF SAMPLE: (Check all that apply)

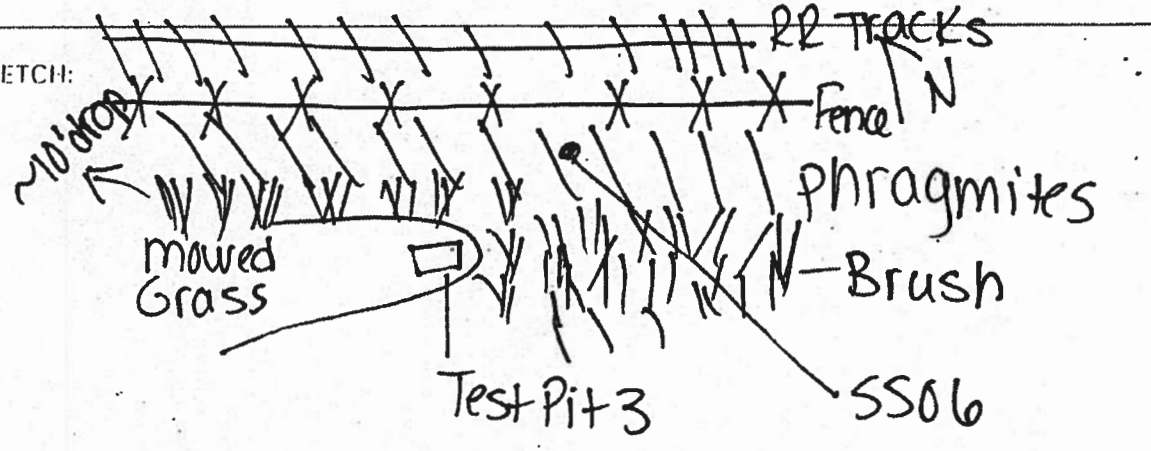
- [X] Soil
- [ ] Sediment
- [ ] Lagoon/Pond
- [ ] Grab
- [ ] Trip Blank*
- [ ] Rinsate Blank*
- [ ] Field Duplicate collected
- [ ] Other (Specify):

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.) ~~Wet~~ Brown - Olive Gray, muck w/ SILT, plant fibers, leaves, roots.

SAMPLE DATA/REMARKS: at Sample location - 4-6" standing Water over Soil.

ANALYSIS	BOTTLE LOT NO.
VOC	
SVOC	
TPH	
Metals	
SVOCs	
Pesticides	
PCB	

NOTES/SKETCH:



Tt NUS Form 0005A

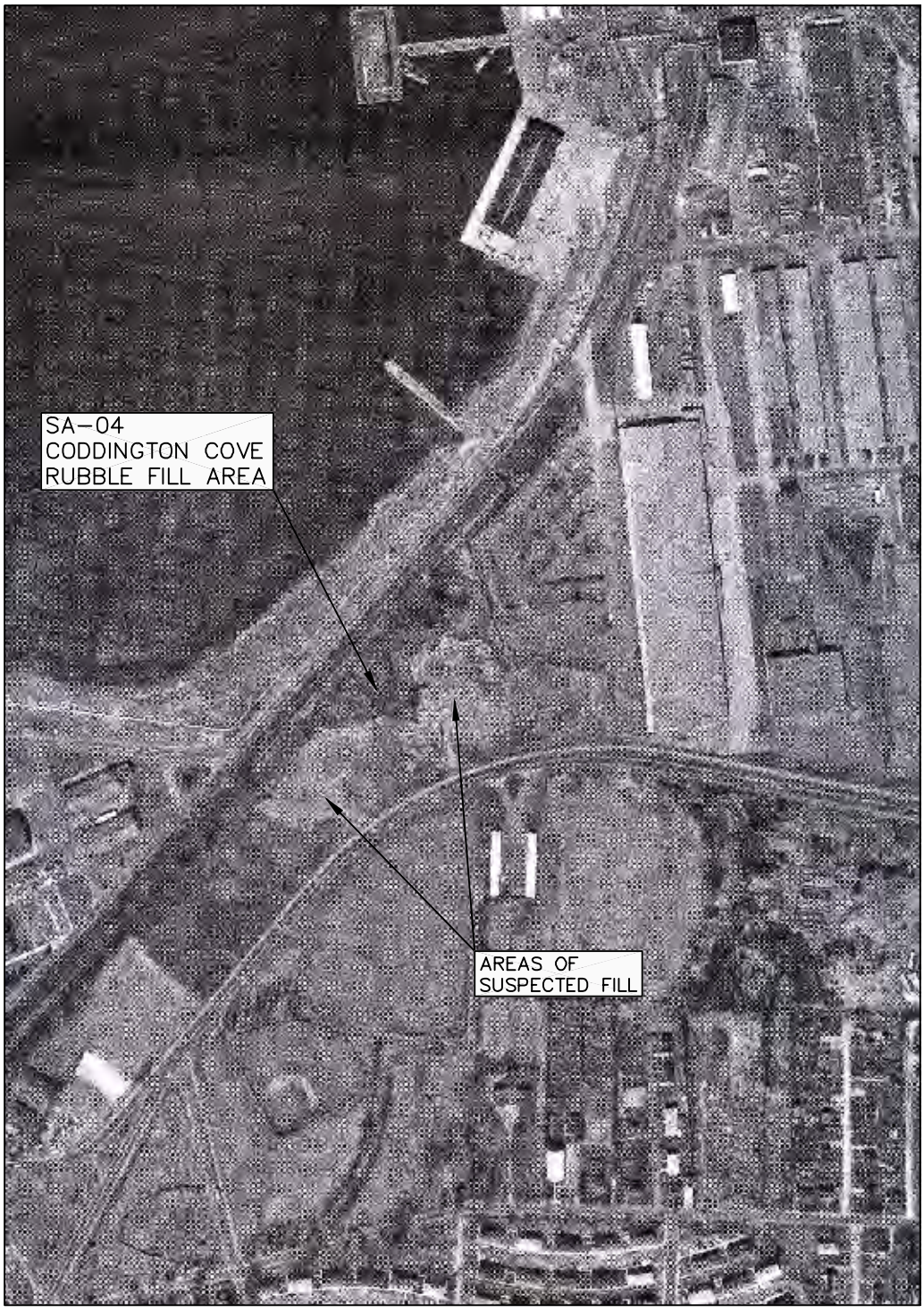
APPENDIX H

HISTORIC AERIAL PHOTOGRAPHS

AERIAL PHOTO – 1951–1952	FIGURE 2–3
CCRF AREA – TECHNICAL MEMORANDUM: RECORD REVIEW AND FIELD SAMPLING PLAN	
NAVSTA NEWPORT – NEWPORT, RHODE ISLAND	

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	L. SEYDEWITZ	DATE:	MAY 3, 2004
SCALE:	NONE	ACAD NAME:	DWG\5152\0471\FIG_2-3.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658–7899



AERIAL PHOTO – 1965

FIGURE 2–4

CCRF AREA – TECHNICAL MEMORANDUM:
RECORD REVIEW AND FIELD SAMPLING PLAN

NAVSTA NEWPORT – NEWPORT, RHODE ISLAND

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	L. SEYDEWITZ	DATE:	MAY 3, 2004
SCALE:	AS NOTED	ACAD NAME:	DWG\5152\0471\FIG_2–4.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658–7899

AERIAL PHOTO – 1981

FIGURE 2–5

CCRF AREA – TECHNICAL MEMORANDUM:
RECORD REVIEW AND FIELD SAMPLING PLAN

NAVSTA NEWPORT – NEWPORT, RHODE ISLAND

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	L. SEYDEWITZ	DATE:	MAY 3, 2004
SCALE:	AS NOTED	ACAD NAME:	DWG\5152\0471\FIG_2–5.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658–7899

AERIAL PHOTO – 1997

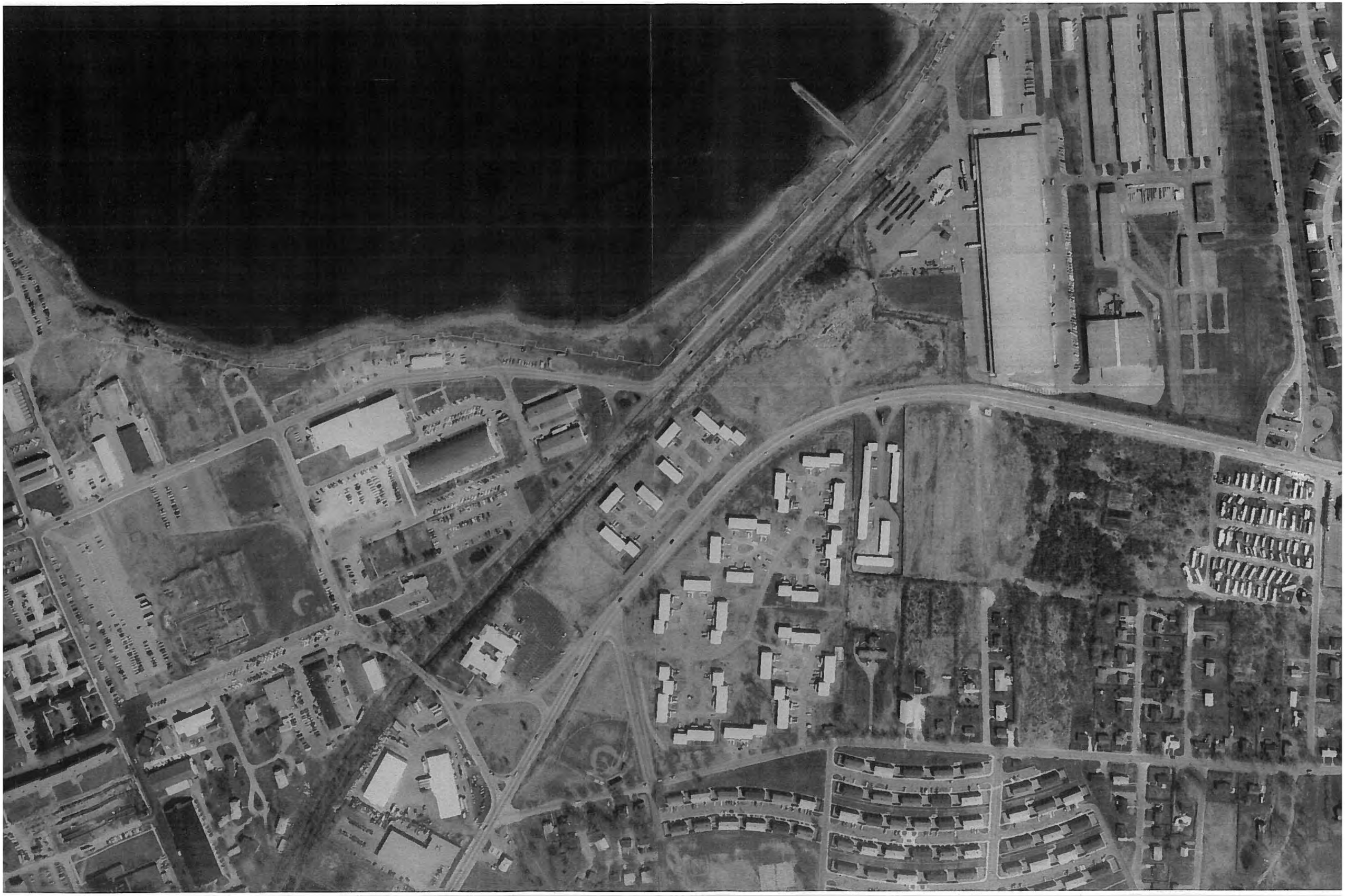
FIGURE 2-6

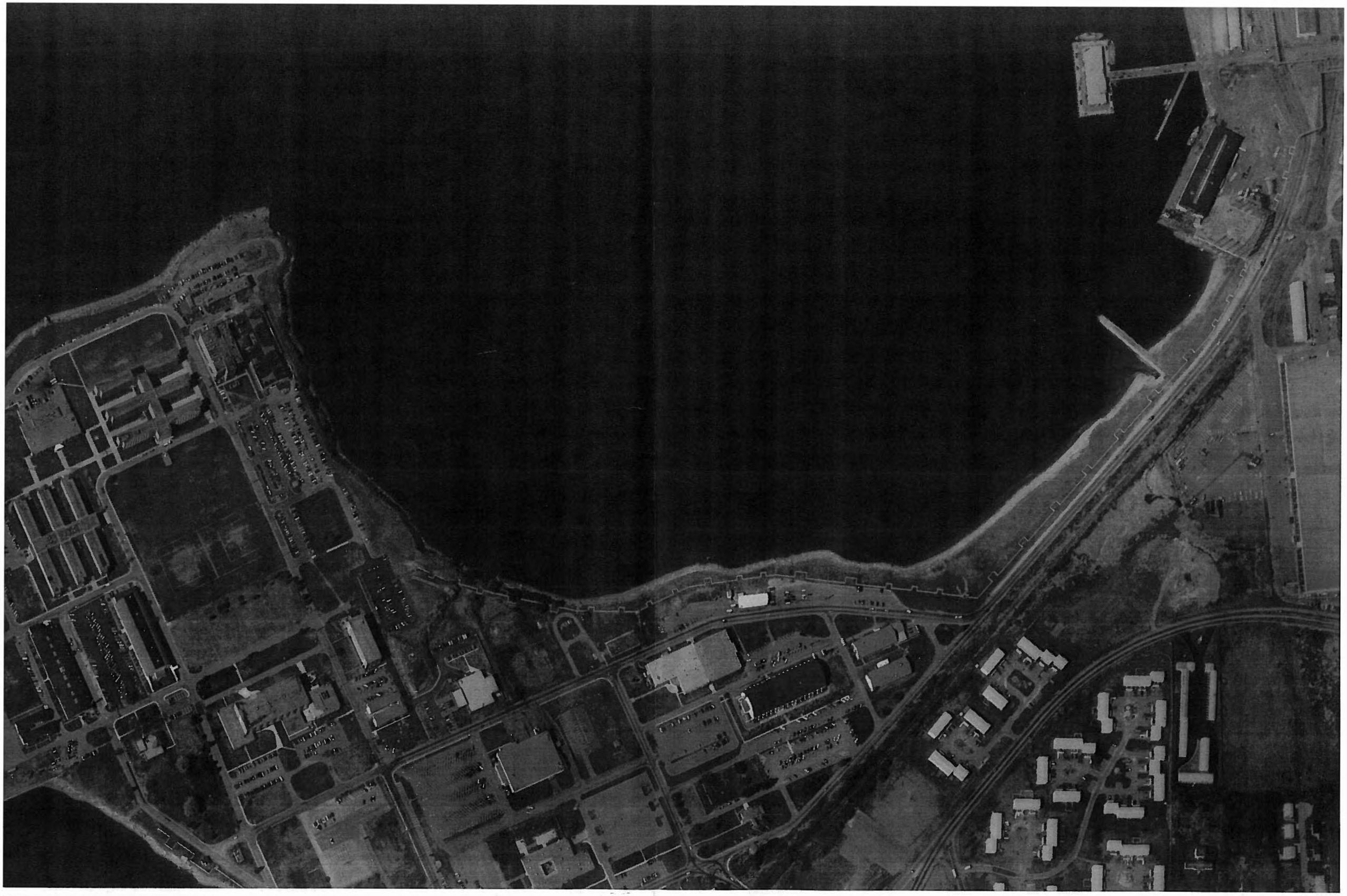
CCRF AREA – TECHNICAL MEMORANDUM:
RECORD REVIEW AND FIELD SAMPLING PLAN

NAVSTA NEWPORT – NEWPORT, RHODE ISLAND

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	L. SEYDEWITZ	DATE:	MAY 3, 2004
SCALE:	AS NOTED	ACAD NAME:	DWG\5152\0471\FIG_2-6.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658-7899





Appendix B

Resolution Consultants SOPs

Utility Clearance

Procedure 3-01

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the process for determining the presence of subsurface utilities and other cultural features at locations where planned site activities involve the physical disturbance of subsurface materials.

1.2 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).

1.3 The procedure applies to the following activities: soil gas surveying, excavating, trenching, drilling of borings and installation of monitoring and extraction wells, use of soil recovery or slide-hammer hand augers, and all other intrusive sampling activities.

1.4 The primary purpose of the procedure is to minimize the potential for damage to underground utilities and other subsurface features, which could result in physical injury, disruption of utility service, or disturbance of other subsurface cultural features.

1.5 If there are procedures, whether it be from Resolution Consultants, state, and/or federal, that are not addressed in this SOP and are applicable to utility clearance, those procedures should be added as an appendix to the project specific SAP.

1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Field and subcontractor personnel shall adhere to a site-specific health and safety plan (HASP).

3.0 Terms and Definitions

3.1 **Utility**

For the proposes of this SOP, a utility is defined as a manmade underground line or conduit, cable, pipe, vault or tank that is, or was, used for the transmission of material or energy (e.g., gas, electrical, telephone, steam, water or sewage, product transfer lines, or underground storage tanks).

3.2 **As-Built Plans**

As-built plans are plans or blueprints depicting the locations of structures and associated utilities on a property.

3.3 **One-Call**

The Utility Notification Center is the one-call agency for nationwide call before you dig. The Utility Notification Center is open 24 hours a day, and accepts calls from anyone planning to dig. The phone number 811 is the designated call before you dig phone number that directly connects you to your local one-call center. Additional information can be found at www.call811.com.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Calling before you dig ensures that any publicly owned underground lines will be marked so that you can dig around them safely. Having the utility lines marked not only prevents accidental damage to the lines, but prevents property damage and personal injuries that could result in breaking a line.

The following information will need to be provided when a call is placed to One-Call:

- Your name, phone number, company name (if applicable), and mailing address.
- What type or work is being done.
- Who the work is being done for.
- The county and city the work is taking place in.
- The address or the street where the work is taking place.
- Marking instructions, (specific instructions as to where the work is taking place).

Under normal circumstances it takes between 2 to 5 days from the time you call (not counting weekends or holidays) to have the underground lines marked. Because these laws vary from state to state, exactly how long it will take depends on where your worksite is located. You will be given an exact start time and date when your locate request is completed, which will comply with the laws in your area.

In the event of an emergency (any situation causing damage to life or property, or a service outage), lines can be marked sooner than the original given time if requested.

3.4 **Toning**

Toning is the process of surveying an area utilizing one or more surface geophysical methods to determine the presence or absence of underground utilities. Typically, toning is conducted after identifying the general location of utilities and carefully examining all available site utility plans. Each location is marked according to the type of utility being identified. In addition, areas cleared by toning are flagged or staked to indicate that all identified utilities in a given area have been toned.

4.0 Training and Qualifications

4.1 The **Contract Task Order (CTO) Manager** is responsible for verifying that these utility locating procedures are performed prior to the initiation of active subsurface exploration.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all utility locating activities are performed in accordance with this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

5.1 Equipment and supplies necessary for locating subsurface utilities will be provided by the subcontractor; however, the project **Field Manager/Field Personnel** will provide any additional equipment and supplies as needed as well as maintain information regarding the utility clearance activities in the field logbook.

6.0 Procedure

Proceed wtih the following steps where subsurface exploration will include excavations, drilling, or any other subsurface investigative method that could damage utilities at a site. In addition to the steps outlined below, always exercise caution while conducting subsurface exploratory work.

6.1 **Prepare Preliminary Site Plan**

- Prepare a preliminary, scaled site plan depicting the proposed exploratory locations as part of the project specific Sampling and Analysis Plan (SAP) or Work Plan. Include as many of the cultural and natural features as practical in this plan.

6.2 **Review Background Information**

- Search existing plan files to review the as-built plans to identify the known location of utilities at the site. Plot the locations of utilities identified onto a preliminary, scaled site plan. Inform the CTO Manager if utilities lie within close proximity to a proposed exploration or excavation location. The CTO Manager will determine if it is necessary to relocate proposed sampling or excavation locations.
- Include the utility location information gathered during previous investigations (e.g., remedial investigation or remedial site evaluation) in the project design documents for removal or remedial actions. In this manner, information regarding utility locations collected during implementation of a CTO can be shared with the subcontractor during implementation of a particular task order. In many instances, this will help to reduce the amount of additional geophysical surveying work the subcontractor may have to perform.
- Conduct interviews with onsite and facility personnel familiar with the site to obtain additional information regarding the known and suspected locations of underground utilities. In addition, if appropriate, contact shall be made with local utility companies to request their help in locating underground lines. Pencil in the dimensions, orientation, and depth of utilities, other than those identified on the as-built plans, at their approximate locations on the preliminary plans. Enter the type of utility, the personnel who provided the information, and the date the information was provided into the field log.
- During the pre-field work interviewing process, the interviewer will determine which site personnel should be notified in the event of an incident involving damage to existing utilities. Record this information in the field logbook with the corresponding telephone numbers and addresses.

6.3 **Site Visit/Locate Utilities/Toning**

- Prior to the initiation of field activities, the Field Task Manager or similarly qualified field personnel shall visit the site and note existing structures and evidence of associated utilities, such as fire hydrants, irrigation systems, manhole and vault box covers, standpipes, telephone switch boxes, free-standing light poles, gas or electric meters, pavement cuts, and linear depression. Compare notes of the actual site configuration to the preliminary site plan. Note deviations in the field logbook and on the preliminary site plan. Accurately locate or survey and clearly mark with stakes, pins, flags, paint, or other suitable devices all areas where subsurface exploration is proposed. These areas shall correspond with the locations drawn on the preliminary site plan.
- Following the initial site visit by the Field Task Manager, a trained utility locating subcontractor will locate, identify, and tone all utilities depicted on the preliminary site plan. The Field Task Manager or similarly qualified field personnel shall visit the site and identify the areas of subsurface disturbance with white spray paint, chalk, white pin flags or some other easily identifiable marking. The utility locator should utilize appropriate sensing equipment to attempt to locate utilities that might not have appeared on the as-built plans. At a minimum, the utility subcontractor should utilize a metal detector and/or magnetometer; however, it is important to consider the possibility that non-metallic utilities or tanks might be present at the site. Use other appropriate surface geophysical methods such as Ground Penetrating Radar, Radiodetection, etc. as appropriate. Clear proposed exploration areas of all utilities in the immediate area where subsurface exploration is proposed. Clearly tone all anomalous areas. Clearly identify all toned areas on the preliminary site plan. All utilities near the area of subsurface disturbance should also be marked out by the utility subcontractor using the universal colors for subsurface utilities (i.e., red – electric; blue – water; green – sewer; yellow – gas; etc.). After toning the site and plotting all known or suspected buried utilities on the preliminary site plan, the utility locator shall provide the Field Task Manager with a copy of the completed preliminary

site plan. Alternatively, the Field Task Manager or designee shall document the results of the survey on the preliminary site plan.

- Report to the Field Task Manager anomalous areas detected and toned that are in close proximity to the exploration or excavation areas. The Field Task Manager shall determine the safe distance to maintain from the known or suspected utility. It may be necessary to relocate the proposed exploration or excavation areas. If this is required, the Field Task Manager or designee shall relocate them and clearly mark them using the methods described above. Completely remove the markings at the prior location. Plot the new locations on the site plan and delete the prior locations from the plan. In some instances, such as in areas extremely congested with subsurface utilities, it may be necessary to dig by hand or use techniques such as air knife to determine the location of the utilities.

6.4 **Prepare Site Plan**

- Prior to the initiation of field activities, draft a final site plan that indicates the location of subsurface exploration areas and all known or suspected utilities present at the site. Provide copies of this site plan to the Navy Technical Representative (NTR), the CTO Manager, and the subcontractor who is to conduct the subsurface exploration/excavation work. Review the site plan with the NTR to verify its accuracy prior to initiating subsurface sampling activities.

7.0 Quality Control and Assurance

7.1 Utility locating must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

8.1 A bound field logbook will be kept detailing all activities conducted during the utility locating procedure.

8.2 The logbook will describe any changes and modifications made to the original exploration plan. The trained utility locator shall prepare a report and keep it in the project file. Also, a copy of the final site plan will be kept in the project file.

9.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual.* Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Caryn DeJesus Senior Scientist	Bob Shoemaker Senior Scientist	Rev 0 – Initial Issue (June 2012)

Logbooks

Procedure 3-02

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the activities and responsibilities pertaining to the identification, use, and control of logbooks and associated field data records.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 In order to keep the logbook clean, store it in a clean location and use it only when outer gloves used for PPE have been removed.

3.0 Terms and Definitions

3.1 **Logbook**

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person assigned responsibility for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 **Data Form**

A data form is a predetermined format utilized for recording field data that may become, by reference, a part of the logbook (e.g., soil boring logs, trenching logs, surface soil sampling logs, groundwater sample logs, and well construction logs are data forms).

4.0 Training and Qualifications

4.1 The **Contract Task Order (CTO) Manager** or **designee** is responsible for determining which team members shall record information in field logbooks and for obtaining and maintaining control of the required logbooks. The **CTO Manager** shall review the field logbook on at least a monthly basis. The **CTO Manager** or **designee** is responsible for reviewing logbook entries to determine compliance with this procedure and to ensure that the entries meet the project requirements.

4.2 A knowledgeable individual such as the **Field Manager**, **CTO Manager**, or **Program Quality Manager** shall perform a technical review of each logbook at a frequency commensurate with the level of activity (weekly is suggested, or, at a minimum, monthly). Document these reviews by the dated signature of the reviewer on the last page or page immediately following the material reviewed.

4.3 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.4 The **Field Manager** is responsible for ensuring that all **field personnel** follow these procedures and that the logbook is completed properly and daily. The **Field Manager** is also responsible for submitting copies to the **CTO Manager**, who is responsible for filing them and submitting a copy (if required by the CTO Statement of Work).

4.5 The **logbook user** is responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature. The **logbook user** is also responsible for safeguarding the logbook while having custody of it.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

4.6 All **field personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

5.1 Field logbooks shall be bound field notebooks with water-repellent pages.

5.2 Pens shall have indelible black ink.

6.0 Procedure

6.1 The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct the applicable events. Store the logbook in a clean location and use it only when outer gloves used for personal protective equipment (PPE) have been removed.

6.2 Individual data forms may be generated to provide systematic data collection documentation. Entries on these forms shall meet the same requirements as entries in the logbook and shall be referenced in the applicable logbook entry. Individual data forms shall reference the applicable logbook and page number. At a minimum, include names of all samples collected in the logbook even if they are recorded elsewhere.

6.3 Enter field descriptions and observations into the logbook, as described in Attachment 1, using indelible black ink.

6.4 Typical information to be entered includes the following:

- Dates (month/day/year) and times (military) of all on-site activities and entries made in logbooks/forms;
- Site name and description;
- Site location by longitude and latitude, if known;
- Weather conditions, including temperature and relative humidity;
- Fieldwork documentation, including site entry and exit times;
- Descriptions of, and rationale for, approved deviations from the work plan (WP) or field sampling plan;
- Field instrumentation readings;
- Names, job functions, and organizational affiliations of on-site personnel;
- Photograph references;
- Site sketches and diagrams made on site;
- Identification and description of sample morphology, collection locations, and sample numbers;
- Sample collection information, including dates (month/day/year) and times (military) of sample collections, sample collection methods and devices, station location numbers, sample collection depths/heights, sample preservation information, sample pH (if applicable), analysis requested (analytical groups), etc., as well as chain-of-custody (COC) information such as sample identification numbers cross-referenced to COC sample numbers;
- Sample naming convention;
- Field quality control (QC) sample information;
- Site observations, field descriptions, equipment used, and field activities accomplished to reconstruct field operations;

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- Meeting information;
- Important times and dates of telephone conversations, correspondence, or deliverables;
- Field calculations;
- PPE level;
- Calibration records;
- Contractor and subcontractor information (address, names of personnel, job functions, organizational affiliations, contract number, contract name, and work assignment number);
- Equipment decontamination procedures and effectiveness;
- Laboratories receiving samples and shipping information, such as carrier, shipment time, number of sample containers shipped, and analyses requested; and
- User signatures.

6.5 The logbook shall reference data maintained in other logs, forms, etc. Correct entry errors by drawing a single line through the incorrect entry, then initialing and dating this change. Enter an explanation for the correction if the correction is more than for a mistake.

6.6 At least at the end of each day, the person making the entry shall sign or initial each entry or group of entries.

6.7 Enter logbook page numbers on each page to facilitate identification of photocopies.

6.8 If a person's initials are used for identification, or if uncommon acronyms are used, identify these on a page at the beginning of the logbook.

6.9 At least weekly and preferably daily, the **preparer** shall photocopy and retain the pages completed during that session for backup. This will prevent loss of a large amount of information if the logbook is lost.

7.0 Quality Control and Assurance

7.1 Review per Section 4.2 shall be recorded.

8.0 Records, Data Analysis, Calculations

8.1 Retain the field logbook as a permanent project record. If a particular CTO requires submittal of photocopies of logbooks, perform this as required.

8.2 Deviations from this procedure shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

9.1 Attachment 1 – Description of Logbook Entries

9.2 Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue

Attachment 1
Description of Logbook Entries

Logbook entries shall be consistent with Section A.1.4 *Field Documentation SOPs* of the UFP-QAPP Manual (DoD 2005) and contain the following information, as applicable, for each activity recorded. Some of these details may be entered on data forms, as described previously.

Name of Activity	For example, Asbestos Bulk Sampling, Charcoal Canister Sampling, Aquifer Testing.
Task Team Members and Equipment	Name all members on the field team involved in the specified activity. List equipment used by serial number or other unique identification, including calibration information.
Activity Location	Indicate location of sampling area as indicated in the field sampling plan.
Weather	Indicate general weather and precipitation conditions.
Level of PPE	Record the level of PPE (e.g., Level D).
Methods	Indicate method or procedure number employed for the activity.
Sample Numbers	Indicate the unique numbers associated with the physical samples. Identify QC samples.
Sample Type and Volume	Indicate the medium, container type, preservative, and the volume for each sample.
Time and Date	Record the time and date when the activity was performed (e.g., 0830/08/OCT/89). Use the 24-hour clock for recording the time and two digits for recording the day of the month and the year.
Analyses	Indicate the appropriate code for analyses to be performed on each sample, as specified in the WP.
Field Measurements	Indicate measurements and field instrument readings taken during the activity.
Chain of Custody and Distribution	Indicate chain-of-custody for each sample collected and indicate to whom the samples are transferred and the destination.
References	If appropriate, indicate references to other logs or forms, drawings, or photographs employed in the activity.
Narrative (including time and location)	Create a factual, chronological record of the team's activities throughout the day including the time and location of each activity. Include descriptions of general problems encountered and their resolution. Provide the names and affiliations of non-field team personnel who visit the site, request changes in activity, impact the work schedule, request information, or observe team activities. Record any visual or other observations relevant to the activity, the contamination source, or the sample itself. It should be emphasized that logbook entries are for recording data and chronologies of events. The logbook author must include observations and descriptive notations, taking care to be objective and recording no opinions or subjective comments unless appropriate.
Recorded by	Include the signature of the individual responsible for the entries contained in the logbook and referenced forms.
Checked by	Include the signature of the individual who performs the review of the completed entries.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Recordkeeping, Sample Labeling, and Chain-of-Custody

Procedure 3-03

1.0 Purpose and Scope

1.1 The purpose of this standard operating procedure is to establish standard protocols for all field personnel for use in maintaining field and sampling activity records, writing sample logs, labeling samples, ensuring that proper sample custody procedures are utilized, and completing chain-of-custody/analytical request forms.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

Not applicable.

3.0 Terms and Definitions

3.1 **Logbook**

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person responsible for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 **Chain-of-Custody**

Chain-of-custody (COC) is documentation of the process of custody control. Custody control includes possession of a sample from the time of its collection in the field to its receipt by the analytical laboratory, and through analysis and storage prior to disposal.

4.0 Training and Qualifications

4.1 The **CTO Manager** is responsible for determining which team members shall record information in the field logbook and for checking sample logbooks and COC forms to ensure compliance with these procedures. The **CTO Manager** shall review COC forms on a monthly basis at a minimum.

4.2 The **CTO Manager** and **Program Quality Manager** are responsible for evaluating project compliance with the Project Procedures Manual.

4.3 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.4 The **Laboratory Project Manager** or **Sample Control Department Manager** is responsible for reporting any sample documentation or COC problems to the **CTO Manager** or **CTO Laboratory Coordinator** within 24 hours of sample receipt.

4.5 The **Field Manager** is responsible for ensuring that all **field personnel** follow these procedures. The **CTO Laboratory Coordinator** is responsible for verifying that the COC/analytical request forms have been completed properly and match the sampling and analysis plan. The **CTO Manager** or **CTO Laboratory Coordinator** is responsible for notifying the **laboratory**, **data managers**, and **data validators** in writing if analytical request changes are required as a corrective action. These small changes are different from change orders, which involve changes to the scope of the subcontract with

the laboratory and must be made in accordance with a respective contract (e.g., CLEAN remedial action contract).

4.6 All **field personnel** are responsible for following these procedures while conducting sampling activities. **Field personnel** are responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature.

5.0 Procedure

This procedure provides standards for documenting field activities, labeling the samples, documenting sample custody, and completing COC/analytical request forms. The standards presented in this section shall be followed to ensure that samples collected are maintained for their intended purpose and that the conditions encountered during field activities are documented.

5.1 Recordkeeping

The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct each day's events. Field logs such as soil boring logs and ground-water sampling logs will also be used. These procedures are described in Procedure 3-02, *Logbooks*.

5.2 Sample Labeling

Affix a sample label with adhesive backing to each individual sample container. Place clear tape over each label (preferably prior to sampling) to prevent the labels from tearing off, falling off, being smeared, and to prevent loss of information on the label. Record the following information with a waterproof marker on each label:

- Project name or number (optional);
- COC sample number;
- Date and time of collection;
- Sampler's initials;
- Matrix (optional);
- Sample preservatives (if applicable); and
- Analysis to be performed on sample (this shall be identified by the method number or name identified in the subcontract with the laboratory).

These labels may be obtained from the analytical laboratory or printed from a computer file onto adhesive labels.

5.3 Custody Procedures

For samples intended for chemical analysis, sample custody procedures shall be followed through collection, transfer, analysis, and disposal to ensure that the integrity of the samples is maintained. Maintain custody of samples in accordance with the U.S. Environmental Protection Agency (EPA) COC guidelines prescribed in EPA *NEIC Policies and Procedures*, National Enforcement Investigations Center, Denver, Colorado, revised May 1986; EPA *RCRA Ground Water Monitoring Technical Enforcement Guidance Document* (TEGD); *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (EPA OSWER Directive 9355 3-01); Appendix 2 of the *Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports*; and *Test Methods for Evaluating Solid Waste* (EPA SW-846)

A description of sample custody procedures is provided below.

5.3.1 Sample Collection Custody Procedures

According to the U.S. EPA guidelines, a sample is considered to be in custody if one of the following conditions is met:

- It is in one's actual physical possession or view;
- It is in one's physical possession and has not been tampered with (i.e., it is under lock or official seal);
- It is retained in a secured area with restricted access; and
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal.

Place custody seals on sample containers immediately after sample collection and on shipping coolers if the cooler is to be removed from the sampler's custody. Place custody seals in such a manner that they must be broken to open the containers or coolers. Label the custody seals with the following information:

- Sampler's name or initials; and
- Date and time that the sample/cooler was sealed.

These seals are designed to enable detection of sample tampering. An example of a custody seal is shown in Attachment 1.

Field personnel shall also log individual samples onto COC forms (carbon copy or computer generated) when a sample is collected. These forms may also serve as the request for analyses. Procedures for completing these forms are discussed in Section 7.4, indicating sample identification number, matrix, date and time of collection, number of containers, analytical methods to be performed on the sample, and preservatives added (if any). The **samplers** will also sign the COC form signifying that they were the personnel who collected the samples. The COC form shall accompany the samples from the field to the laboratory. When a cooler is ready for shipment to the analytical laboratory, the **person delivering the samples for transport** will sign and indicate the date and time on the accompanying COC form. One copy of the COC form will be retained by the **sampler** and the remaining copies of the COC form shall be placed inside a self-sealing bag and taped to the inside of the cooler. Each cooler must be associated with a unique COC form. Whenever a transfer of custody takes place, **both parties** shall sign and date the accompanying carbon copy COC forms, and the **individual relinquishing the samples** shall retain a copy of each form. One exception is when the samples are shipped; the **delivery service personnel** will not sign or receive a copy because they do not open the coolers. The **laboratory** shall attach copies of the completed COC forms to the reports containing the results of the analytical tests. An example COC form is provided in Attachment 2.

5.3.2 Laboratory Custody Procedures

The following custody procedures are to be followed by an **independent laboratory** receiving samples for chemical analysis; the procedures in their Naval Facilities Engineering Service Center-evaluated Laboratory Quality Assurance Plan must follow these same procedures. A **designated sample custodian** shall take custody of all samples upon their arrival at the analytical laboratory. The **custodian** shall inspect all sample labels and COC forms to ensure that the information is consistent, and that each is properly completed. The **custodian** will also measure the temperature of the temperature blank in the coolers upon arrival using either a National Institute for Standards and Technology calibrated thermometer or an infra-red temperature gun. The **custodian** shall note the condition of the samples including:

- If the samples show signs of damage or tampering;
- If the containers are broken or leaking;
- If headspace is present in sample vials;
- If proper preservation of samples has occurred (made by pH measurement, except volatile organic compounds [VOCs] and purgeable total petroleum hydrocarbons [TPH] and temperature). The pH of VOC and purgeable TPH samples will be checked by the **laboratory analyst** after the sample aliquot has been removed from the vial for analysis; and
- If any sample holding times have been exceeded.

All of the above information shall be documented on a sample receipt sheet by the **custodian**.

Discrepancies or improper preservation shall be noted by the **laboratory** as an out-of-control event and shall be documented on an out-of-control form with corrective action taken. The out-of-control form shall be signed and dated by the **sample control custodian** and **any other persons** responsible for corrective action. An example of an out-of-control form is included as Attachment 4.

The **custodian** shall then assign a unique laboratory number to each sample and distribute the samples to secured storage areas maintained at 4 degrees Celsius (soil samples for VOC analysis are to be stored in a frozen state until analysis). The unique laboratory number for each sample, COC sample number, client name, date and time received, analysis due date, and storage shall also be manually logged onto a sample receipt record and later entered into the laboratory's computerized data management system. The **custodian** shall sign the shipping bill and maintain a copy.

Laboratory personnel shall be responsible for the care and custody of samples from the time of their receipt at the laboratory through their exhaustion or disposal. Samples should be logged in and out on internal laboratory COC forms each time they are removed from storage for extraction or analysis.

5.4 Completing COC/Analytical Request Forms

COC form/analytical request form completion procedures are crucial in properly transferring the custody and responsibility of samples from field personnel to the laboratory. This form is important for accurately and concisely requesting analyses for each sample; it is essentially a release order from the analysis subcontract.

Attachment 2 is an example of a generic COC/analytical request form that may be used by **field personnel**. Multiple copies may be tailored to each project so that much of the information described below need not be handwritten each time. Attachment 3 is an example of a completed site-specific COC/analytical request form, with box numbers identified and discussed in text below.

COC forms tailored to each CTO can be drafted and printed onto multi-ply forms. This eliminates the need to rewrite the analytical methods column headers each time. It also eliminates the need to write the project manager, name, and number; QC Level; TAT; and the same general comments each time.

Complete one COC form per cooler. Whenever possible, place all VOC analyte vials into one cooler in order to reduce the number of trip blanks. Complete all sections and be sure to sign and date the COC form. One copy of the COC form must remain with the field personnel.

Box 2 **Bill To:** List the name and address of the person/company to bill only if it is not in the subcontract with the laboratory.

Box 3 **Sample Disposal Instructions:** These instructions will be stated in the Master Service Agreement or each CTO statement of work with each laboratory.

Shipment Method: State the method of shipment (e.g., hand carry or air courier via FedEx or DHL).

Comments: This area shall be used by the field team to communicate observations, potential hazards, or limitations that may have occurred in the field or additional information regarding analysis (e.g., a specific metals list, samples expected to contain high analyte concentrations).

Box 4 **Cooler No.:** This will be written on the inside or outside of the cooler and shall be included on the COC. Some laboratories attach this number to the trip blank identification, which helps track samples for VOC analysis. If a number is not on the cooler, field personnel shall assign a number, write it on the cooler, and write it on the COC.

QC Level: Enter the reporting quality control (QC) requirements (e.g., Full Data Package, Summary Data Package).

Turnaround time (TAT): TAT will be determined by a sample delivery group (SDG), which may be formed over a 14-day period, not to exceed 20 samples. Once the SDG has been completed, standard TAT is 21 calendar days from receipt of the last sample in the SDG. Entering NORMAL or STANDARD in this field will be acceptable. If quicker TAT is required, it shall be in the subcontract with the laboratory and reiterated on each COC to remind the laboratory.

Box 5 **Type of Containers:** Write the type of container used (e.g., 1-liter glass amber, for a given parameter in that column).

Preservatives: Field personnel must indicate on the COC the correct preservative used for the analysis requested. Indicate the pH of the sample (if tested) in case there are buffering conditions found in the sample matrix.

Box 6 **Sample Identification (ID) Number:** This is typically a five-character alphanumeric identifier used by the contractor to identify samples. The use of this identifier is important since the laboratories are restricted to the number of characters they are able to use. Sample numbering shall be in accordance with the project-specific sampling and analysis plan.

Description (Sample ID): This name will be determined by the location and description of the sample, as described in the project-specific sampling and analysis plan. This sample identification should not be submitted to the laboratory, but should be left blank. If a computer COC version is used, the sample identification can be input, but printed with this block black. A cross-referenced list of the COC Sample Number and sample identification must be maintained separately.

Date Collected: Record the collection date in order to track the holding time of the sample. Note: For trip blanks, record the date it was placed in company with samples.

Time Collected: When collecting samples, record the time the sample is first collected. Use of the 24-hour military clock will avoid a.m. or p.m. designations (e.g., 1815 instead of 6:15 p.m.). Record local time; the laboratory is responsible for calculating holding times to local time.

Lab ID: This is for laboratory use only.

Box 7 **Matrix/QC:** Identify the matrix (e.g., water, soil, air, tissue, fresh water sediment, marine sediment, or product). If a sample is expected to contain high analyte concentrations (e.g., a tank bottom sludge or distinct product layer), notify the laboratory in the comment section. Mark an "X" for the sample(s) that have extra volume for laboratory QC matrix spike/matrix spike duplicate (MS/MSD) purposes. The sample provided for MS/MSD purposes is usually a field duplicate.

Box 8 **Analytical Parameters:** Enter the parameter by descriptor and the method number desired (e.g., BTEX 8260B, PAHs 8270C, etc.). Whenever practicable, list the parameters as they appear in the laboratory subcontract to maintain consistency and avoid confusion.

If the COC does not have a specific box for number of sample containers, use the boxes below the analytical parameter, to indicate the number of containers collected for each parameter.

Box 9 **Sampler's Signature:** The person who collected samples must sign here.

Relinquished By: The person who turned over the custody of the samples to a second party other than an express mail carrier, such as FedEx or DHL, must sign and date here.

Received By: Typically, a representative of the receiving laboratory signs and dates here. Or, a field crew member who delivered the samples in person from the field to the laboratory might sign here. A courier, such as FedEx or DHL, does not sign here because they do not open the coolers. It must also be used by the prime contracting laboratory when samples are to be sent to a subcontractor.

Relinquished By: In the case of subcontracting, the primary laboratory will sign and date the Relinquished By space and fill out an additional COC to accompany the samples being subcontracted.

Received By (Laboratory): This space is for the final destination (e.g., at a subcontracted laboratory). A representative of the final destination (e.g., subcontracted laboratory) must sign and date here.

Box 10 **Lab No. and Questions:** This box is to be filled in by the laboratory only.

Box 11 **Control Number:** This number is the "COC" followed by the first contractor identification number in that cooler, or contained on that COC. This control number must be unique (i.e., never used twice). Record the date the COC is completed. It should be the same date the samples are collected.

Box 12 **Total # of Containers:** Sum the number of containers in that row.

Box 13 **Totals:** Sum the number of containers in each column. Because COC forms contain different formats depending on who produced the form, not all of the information listed in items 1 to 13 may be recorded; however, as much of this information as possible shall be included.

6.0 Quality Control and Assurance

6.1 Recordkeeping, sample labeling, and chain-of-custody activities must incorporate quality control measures to ensure accuracy and completeness.

6.2 Deviations from this procedure or the project-specific CTO work plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

7.0 Records, Data Analysis, Calculations

7.1 The COC/analytical request form shall be faxed approximately daily to the **CTO Laboratory Coordinator** for verification of accuracy. Following the completion of sampling activities, the sample

logbook and COC forms will be transmitted to the **CTO Manager** for storage in project files. The **data validators** shall receive a copy also. The original COC/analytical request form shall be submitted by the **laboratory** along with the data delivered. Any changes to the analytical requests that are required shall be made in writing to the laboratory. A copy of this written change shall be sent to the data validators and placed in the project files. The reason for the change shall be included in the project files so that recurring problems can be easily identified.

7.2 Deviations from this procedure or the project-specific sampling and analysis plan shall be documented in the records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or References

8.1 Attachment 1 – Chain-of-Custody Seal

8.2 Attachment 2 – Generic Chain-of-Custody/Analytical Request Form

8.3 Attachment 3 – Sample Completed Chain-of-Custody

8.4 Attachment 4 – Sample Out-of-Control Form

8.5 Environmental Protection Agency, United States (EPA). 1988. *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA.* Interim Final. EPA/540/G-89/004. Office of Emergency and Remedial Response. October.

8.6 EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

8.7 EPA. 1997. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846. 3rd ed., Final Update IIIA. Office of Solid Waste.

8.8 Water Resources Control Board, State of California. 1988. *Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports*. August.

8.9 Procedure 3-02, *Logbooks.*

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 1
Chain-of-Custody Seal

CHAIN-OF-CUSTODY SEAL

[LABORATORY]	SAMPLE NO.	DATE	SEAL BROKEN BY
	SIGNATURE		DATE
	PRINT NAME AND TITLE *(Inspector, Analyst or Technician*		

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 2
Generic Chain-of-Custody / Analytical Request Form

M601376

CHAIN OF CUSTODY RECORD Page ____ of ____

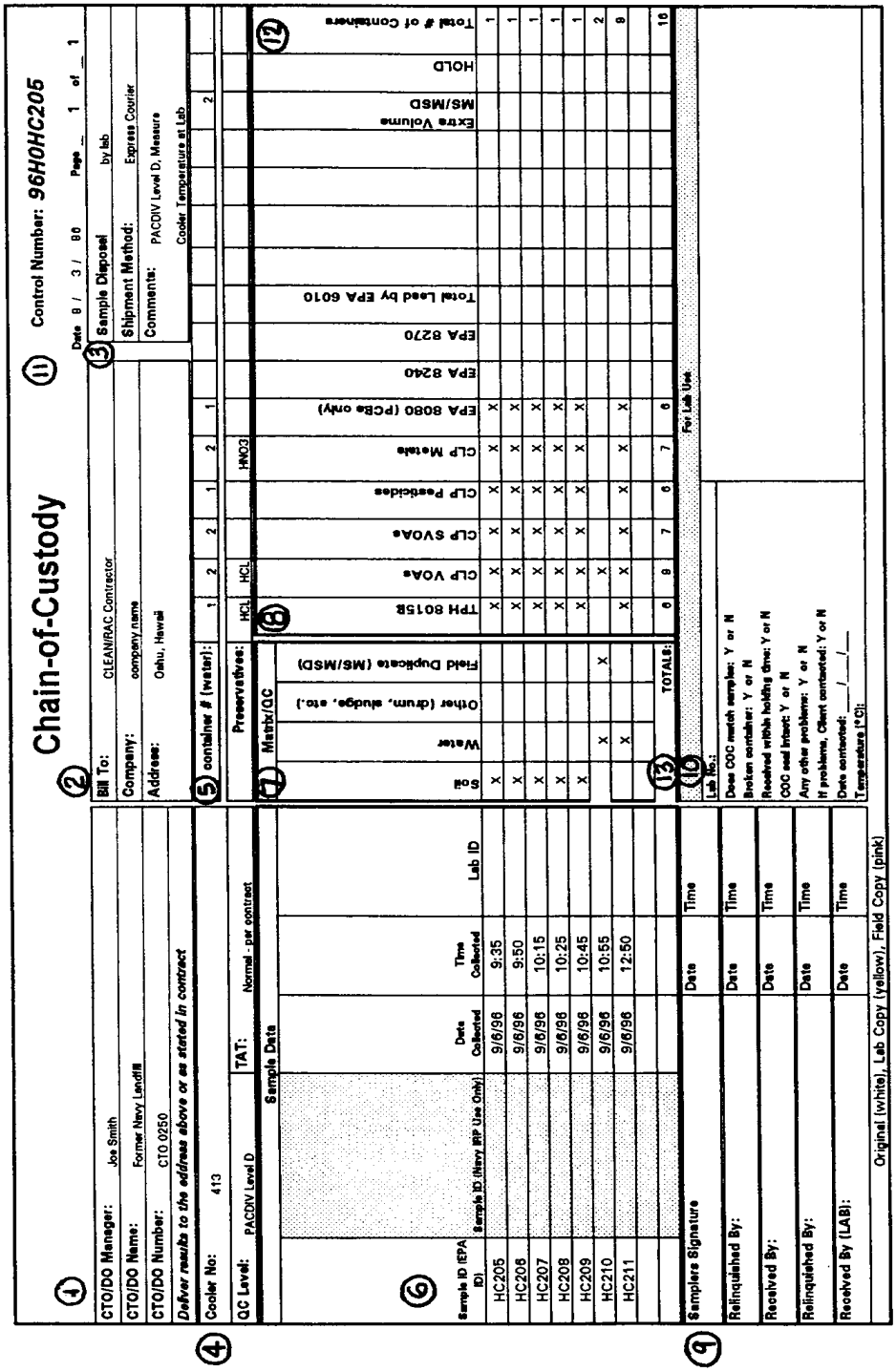
Client/Project Name:	Project Location:	Analysis Requested
Project Number:	Field Logbook No.:	
Sampler: (Print Name) /Affiliation:	Chain of Custody Tape No.:	
Signature:	Send Results/Report to:	

Field Sample No./ Identification	Date	Time	Grab	Comp	Sample Container (Size/Mat'l)	Sample Type (Liquid, Sludge, Etc.)	Preservative	Field Filtered							Lab I.D.	Remarks

Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	Analytical Laboratory (Destination):
Signature:	Time:	Signature:	Time:	
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	
Signature:	Time:	Signature:	Time:	
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:	
Signature:	Time:	Signature:	Time:	Serial No.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 3
Sample Completed Chain-of-Custody



PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 4
Sample Out-of-Control Form

OUT OF CONTROL FORM	Status	Date	Initial
	Noted OOC		
	Submit for CA*		
	Resubmit for CA*		
	Completed		

Date Recognized:	By:
Dated Occurred:	Matrix
Parameter (Test Code):	Method:
Analyst:	Supervisor:

Samples Affected (List by Accession AND Sample No.)

1. Type of Event (Check all that apply)		2. Corrective Action (CA)* (Check all that apply)	
	Calibration Corr. Coefficient <0.995		Repeat calibration
	%RSD>20%		Made new standards
	Blank >MDL		Reran analysis
	Does not meet criteria:		Sample(s) redigested and rerun
	Spike		Sample(s) reextracted and rerun
	Duplicate		Recalculated
	LCS		Cleaned system
	Calibration Verification		Ran standard additions
	Standard Additions		Notified
	MS/MSD		Other (please explain)
	BS/BSD		
	Surrogate Recovery		
	Calculations Error		
	Holding Times Missed		
	Other (Please explain	Comments:	

3. Results of Corrective Action	
	Return to Control (indicated with)
	Corrective Actions Not Successful - DATA IS TO BE FLAGGED with _____.

Analyst:	Date:
Supervisor:	Date:
QA Department:	Date:

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Sample Handling, Storage, and Shipping

Procedure 3-04

1.0 Purpose and Scope

1.1 This standard operating procedure describes the actions to be used by personnel engaged in handling, storing, and transporting samples. The objective is to obtain samples of actual conditions with as little alteration as possible.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Avoid lifting heavy coolers with back muscles; instead, use leg muscles or dollies.

2.2 Wear proper gloves, such as blue nitrile and latex, as defined in the project-specific health and safety plan, when handling sample containers to avoid contacting any materials that may have spilled out of the sample containers.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **Contract Task Order (CTO) Manager** and the **Laboratory Project Manager** are responsible for identifying instances of non-compliance with this procedure and ensuring that future sample transport activities comply with this procedure.

4.2 The **Field Manager** is responsible for ensuring that all samples are shipped according to this procedure.

4.3 **Field personnel** are responsible for the implementation of this procedure.

4.4 The **Program Quality Manager** is responsible for ensuring that sample handling, storage, and transport activities conducted during all CTOs comply with this procedure.

4.5 All **field personnel** are responsible for the implementation of this procedure.

5.0 Procedure

5.1 **Handling and Storage**

Immediately following collection, label all samples according to Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*. The lids of the containers shall not be sealed with duct tape, but may be covered with custody seals or placed directly into self-sealing bags. Place the sample containers in an insulated cooler with frozen gel packs (e.g., "blue ice") or ice in double, sealed self-sealing bags. Samples should occupy the lower portion of the cooler, while the ice should occupy the upper portion. Place an absorbent material (e.g., proper absorbent cloth material) on the bottom of the cooler to contain liquids in case of spillage. Fill all empty space between sample containers with Styrofoam® "peanuts" or other appropriate material. Prior to shipping, wrap glass sample containers on the sides, tops, and bottoms with bubble wrap or other appropriate padding and/or surround them in Styrofoam to

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

prevent breakage during transport. Pack all glass containers for water samples in an upright position, never stacked or on their sides. Prior to shipment, replace the ice or cold packs in the coolers so that samples will be maintained as close to 4 degrees Celsius (°C) as possible from the time of collection through transport to the analytical laboratory. Ship samples within 24 hours or on a schedule allowing the laboratory to meet holding times for analyses. The procedures for maintaining sample temperatures at 4°C pertain to all field samples.

5.2 Shipping

Follow all appropriate U.S. Department of Transportation regulations (e.g., 49 Code of Federal Regulations [CFR], Parts 171-179) for shipment of air, soil, water, and other samples. Elements of these procedures are summarized below.

5.2.1 Hazardous Materials Shipment

Field personnel must state whether any sample is suspected to be a hazardous material. A sample should be assumed hazardous unless enough evidence exists to indicate it is non-hazardous. If not suspected to be hazardous, shipments may be made as described in the Section 5.2.2 for non-hazardous materials. If hazardous, follow the procedures summarized below.

Any substance or material that is capable of posing an unreasonable risk to life, health, or property when transported is classified as hazardous. Perform hazardous materials identification by checking the list of dangerous goods for that particular mode of transportation. If not on that list, materials can be classified by checking the Hazardous Materials Table (49 CFR 172.102 including Appendix A) or by determining if the material meets the definition of any hazard class or division (49 CFR Part 173), as listed in Attachment 2.

All **persons shipping hazardous materials** must be properly trained in the appropriate regulations, as required by HM-126F, Training for Safe Transportation of Hazardous Materials (49 CFR HM-126F Subpart H). The training covers loading, unloading, handling, storing, and transporting of hazardous materials, as well as emergency preparedness in the case of accidents. **Carriers**, such as commercial couriers, must also be trained. Modes of shipment include air, highway, rail, and water.

When shipping hazardous materials, including bulk chemicals or samples suspected of being hazardous, the proper shipping papers (49 CFR 172 Subpart C), package marking (49 CFR 172 Subpart D), labeling (49 CFR 172 Subpart E), placarding (49 CFR 172 Subpart F, generally for carriers), and packaging must be used. Attachment 1 shows an example of proper package markings. Refer to a copy of 49 CFR each time hazardous materials/potentially hazardous samples are shipped.

According to Section 2.7 of the International Air Transport Association Dangerous Goods Regulations publication, very small quantities of certain dangerous goods may be transported without certain marking and documentation requirements as described in 49 CFR Part 172; however, other labeling and packing requirements must still be followed. Attachment 2 shows the volume or weight for different classes of substances. A "Dangerous Goods in Excepted Quantities" label must be completed and attached to the associated shipping cooler (Attachment 3). Certain dangerous goods are not allowed on certain airlines in any quantity.

As stated in item 4 of Attachment 4, the Hazardous Materials Regulations do not apply to hydrochloric acid (HCl), nitric acid (HNO_3), sulfuric acid (H_2SO_4), and sodium hydroxide (NaOH) added to water samples if their pH or percentage by weight criteria is met. These samples may be shipped as non-hazardous materials as discussed below.

5.2.2 Non-Hazardous Materials Shipment

If the samples are suspected to be non-hazardous based on previous site sample results, field screening results, or visual observations, if applicable, then samples may be shipped as non-hazardous.

When a cooler is ready for shipment to the laboratory, place two copies of the chain-of-custody form inside a self-sealing bag and tape it to the inside of the insulated cooler. Then, seal the cooler with waterproof tape and label it with "Fragile," "This-End-Up" (or directional arrows pointing up), or other appropriate notices. Place chain-of-custody seals on the coolers as discussed in Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*.

5.2.3 Shipments from Outside the Continental United States

Shipment of sample coolers to the United States from locations outside the continental United States is controlled by the U.S. Department of Agriculture (USDA) and is subject to their inspection and regulation. A "USDA Soil Import Permit" is required to prove that the receiving analytical laboratory is certified by the USDA to receive and properly dispose of soil. In addition, all sample coolers must be inspected by a **USDA representative**, affixed with a label indicating that the coolers contain environmental samples, and accompanied by shipping forms stamped by the **USDA inspector** prior to shipment.

In addition, the U.S. Customs Service must clear samples shipped from U.S. territorial possessions or foreign countries upon entry into the United States. As long as the commercial invoice is properly completed (see below), shipments typically pass through U.S. Customs Service without the need to open coolers for inspection.

Completion and use of proper paperwork will, in most cases, minimize or eliminate the need for the USDA and U.S. Customs Service to inspect the contents. Attachment 5 shows an example of how paperwork may be placed on the outside of coolers for non-hazardous materials. For hazardous materials, refer to Section 5.2.1.

In summary, tape the paperwork listed below to the outside of the coolers to accompany sample shipments. If a shipment is made up of multiple pieces (e.g., more than one cooler), the paperwork need only be attached to one cooler, provided that the **courier** agrees. All other coolers in the shipment need only to be taped and have the address and chain-of-custody seals affixed.

1. **Courier Shipping Form & Commercial Invoice:** See Attachment 6 and Attachment 7 for examples of the information to be included on the commercial invoices for soil and water, respectively. Place the courier shipping form and commercial invoice inside a clear, plastic, adhesive-backed pouch that adheres to the package (typically supplied by the courier) and place it on the cooler lid as shown in Attachment 5.
2. **Soil Import Permit (soil only):** See Attachment 8 and Attachment 9 for examples of the soil import permit and soil samples restricted entry labels, respectively. The **laboratory** shall supply these documents prior to mobilization. The USDA often stops shipments of soil without these documents. Staple together the 2-inch × 2-inch USDA label (described below) and soil import permit, and place them inside a clear plastic pouch. The **courier** typically supplies the clear, plastic, adhesive-backed pouches that adhere to the package.

   Placing one restricted entry label as shown in Attachment 5 (covered with clear packing tape) and one stapled to the actual permit is suggested.

   The USDA does not control water samples, so the requirements for soil listed above do not apply.
3. **Chain-of-Custody Seals:** The **laboratory** should supply the seals. **CTO personnel** must sign and date these. At least two seals should be placed in such a manner that they stick to both the cooler lid and body. Placing the seals over the tape (as shown in Attachment 5), then covering it with clear packing tape is suggested. This prevents the seal from coming loose and enables detection of tampering.
4. **Address Label:** Affix a label stating the destination (laboratory address) to each cooler.
5. **Special Requirements for Hazardous Materials:** See Section 5.2.1.

Upon receipt of sample coolers at the laboratory, the **sample custodian** shall inspect the sample containers as discussed in Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*. The samples shall then be immediately extracted and/or analyzed, or stored in a refrigerated storage area until they are removed for extraction and/or analysis. Whenever the samples are not being extracted or analyzed, they shall be returned to refrigerated storage.

6.0 Quality Control and Assurance

6.1 Sample handling, storage, and shipping must incorporate quality control measures to ensure conformance to these and the project requirements.

7.0 Records, Data Analysis, Calculations

7.1 Maintain records as required by implementing these procedures.

7.2 Deviations from this procedure or the project-specific sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or Reference

8.1 Attachment 1 – Example Hazardous Material Package Marking

8.2 Attachment 2 – Packing Groups

8.3 Attachment 3 – Label for Dangerous Goods in Excepted Quantities

8.4 Attachment 4 – SW-846 Preservative Exception

8.5 Attachment 5 – Non-Hazardous Material Cooler Marking Figure for Shipment from Outside the Continental United States

8.6 Attachment 6 – Commercial Invoice – Soil

8.7 Attachment 7 – Commercial Invoice – Water

8.8 Attachment 8 – Soil Import Permit

8.9 Attachment 9 – Soil Samples Restricted Entry Labels

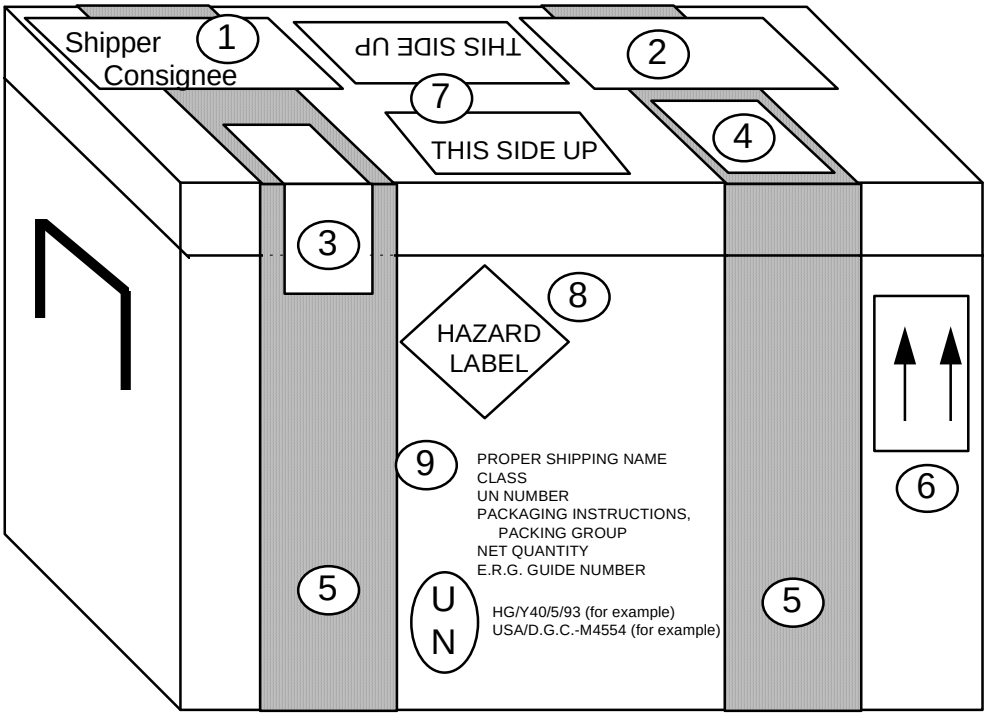
8.10 NAVSEA T0300-AZ-PRO-010. *Navy Environmental Compliance Sampling and Field Testing Procedures Manual*. August 2009.

8.11 Procedure 3-03, *Recordkeeping, Sample Labeling, and Chain-of-Custody*.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 1
Example Hazardous Material Package Marking



1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**
7. **THIS SIDE UP STICKERS**
8. **HAZARD LABEL**
9. **HAZARDOUS MATERIAL INFORMATION**
10. **PACKAGE SPECIFICATIONS**

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 2
Packing Groups

PACKING GROUP OF THE SUBSTANCE	PACKING GROUP 1		PACKING GROUP II		PACKING GROUP III	
CLASS or DIVISION of PRIMARY or SUBSIDIARY RISK	Packagings		Packagings		Packagings	
	Inner	Outer	Inner	Outer	Inner	Outer
1: Explosives	---------- Forbidden (Note A) ----------					
2.1: Flammable Gas	---------- Forbidden (Note B) ----------					
2.2: Non-Flammable, non-toxic gas	---------- See Notes A and B ----------					
2.3: Toxic gas	---------- Forbidden (Note A) ----------					
3. Flammable liquid	30 mL	300 mL	30 mL	500 mL	30 mL	1 L
4.1 Self-reactive substances	Forbidden		Forbidden		Forbidden	
4.1: Other flammable solids	Forbidden		30 g	500 g	30 g	1 kg
4.2: Pyrophoric substances	Forbidden		Not Applicable		Not Applicable	
4.2 Spontaneously combustible substances	Not Applicable		30 g	500 g	30 g	1 kg
4.3: Water reactive substances	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.1: Oxidizers	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.2: Organic peroxides (Note C)	See Note A		30 g or 30 mL	500 g or 250 mL	Not Applicable	
6.1: Poisons - Inhalation toxicity	Forbidden		1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1: Poisons - oral toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1: Poisons - dermal toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.2: Infectious substances	---------- Forbidden (Note A) ----------					
7: Radioactive material (Note D)	---------- Forbidden (Note A) ----------					
8: Corrosive materials	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
9: Magnetized materials	---------- Forbidden (Note A) ----------					
9: Other miscellaneous materials (Note E)	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L

Note A: *Packing groups are not used for this class or division.*
Note B: *For inner packagings, the quantity contained in receptacle with a water capacity of 30 mL. For outer packagings, the sum of the water capacities of all the inner packagings contained must not exceed 1 L.*
Note C: *Applies only to Organic Peroxides when contained in a chemical kit, first aid kit or polyester resin kit.*
Note D: *See 6.1.4.1, 6.1.4.2, and 6.2.1.1 through 6.2.1.7, radioactive material in excepted packages.*
Note E: *For substances in Class 9 for which no packing group is indicated in the List of Dangerous Goods, Packing Group II quantities must be used.*

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 3
Dangerous Goods in Excepted Quantities

DANGEROUS GOODS IN EXCEPTED QUANTITIES

This package contains dangerous goods in excepted small quantities and is in all respects in compliance with the applicable international and national government regulations and the IATA Dangerous Goods Regulations.

_____

Signature of Shipper

_____ _____

Title Date

_____

_____

Name and address of Shipper

This package contains substance(s) in Class(es)
(check applicable box(es))

Class:	2	3	4	5	6	8	9
	☐	☐	☐	☐	☐	☐	☐

and the applicable UN Numbers are:

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 4
SW-846 Preservative Exception

Measurement	Vol. Req. (mL)	Container[2]	Preservative[3,4]	Holding Time[5]
MBAS	250	P, G	Cool, 4ºC	48 Hours
NTA	50	P, G	Cool, 4ºC	24 Hours

1. More specific instructions for preservation and sampling are found with each procedure as detailed in this manual. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, p. 72-82 (1976) Method D-3370.

2. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.

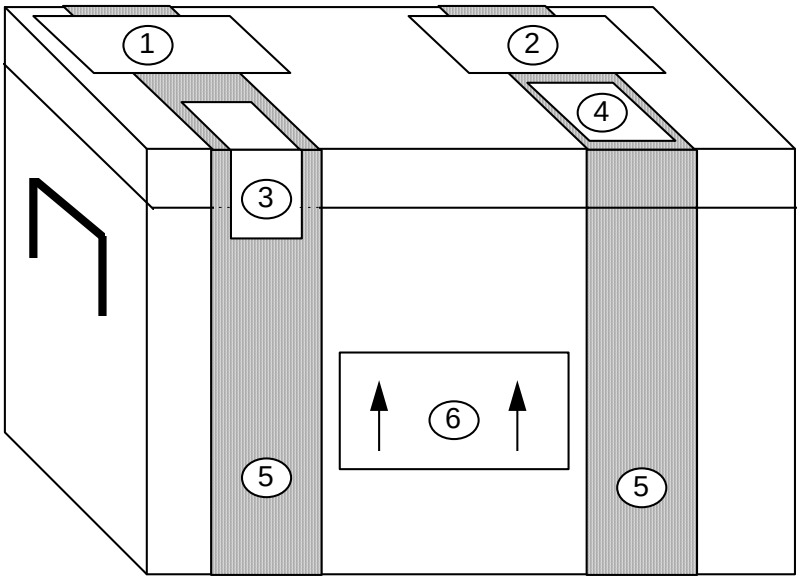
3. Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4ºC until compositing and sample splitting is completed.

4. When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. for the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentration of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

5. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of sample under study are stable for the longer time, and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.

6. Should only be used in the presence of residual chlorine.

Attachment 5
Non-Hazardous Material Cooler Marking Figure for Shipment from Outside the Continental United States



1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**

Attachment 6
Commercial Invoice – Soil

DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <CTO #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	
INTERNATIONAL AIR WAYBILL NO.	(NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Soil samples for laboratory analysis only				$1.00	$3.00

TOTAL NO. OF PKGS.	TOTAL WEIGHT	TOTAL INVOICE VALUE
3		$3.00

Check one
☐ F.O.B.
☐ C&F
☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.

DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT

SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden	Joe Smith	1/1/94
Name/Title	Signature	Date

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 7
Commercial Invoice – Water

DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <COC #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	

INTERNATIONAL AIR WAYBILL NO. _____ (NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Water samples for laboratory analysis only				$1.00	$3.00
	TOTAL NO. OF PKGS.					TOTAL WEIGHT		TOTAL INVOICE VALUE
	3							$3.00

Check one
☐ F.O.B.
☐ C&F
☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.

DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

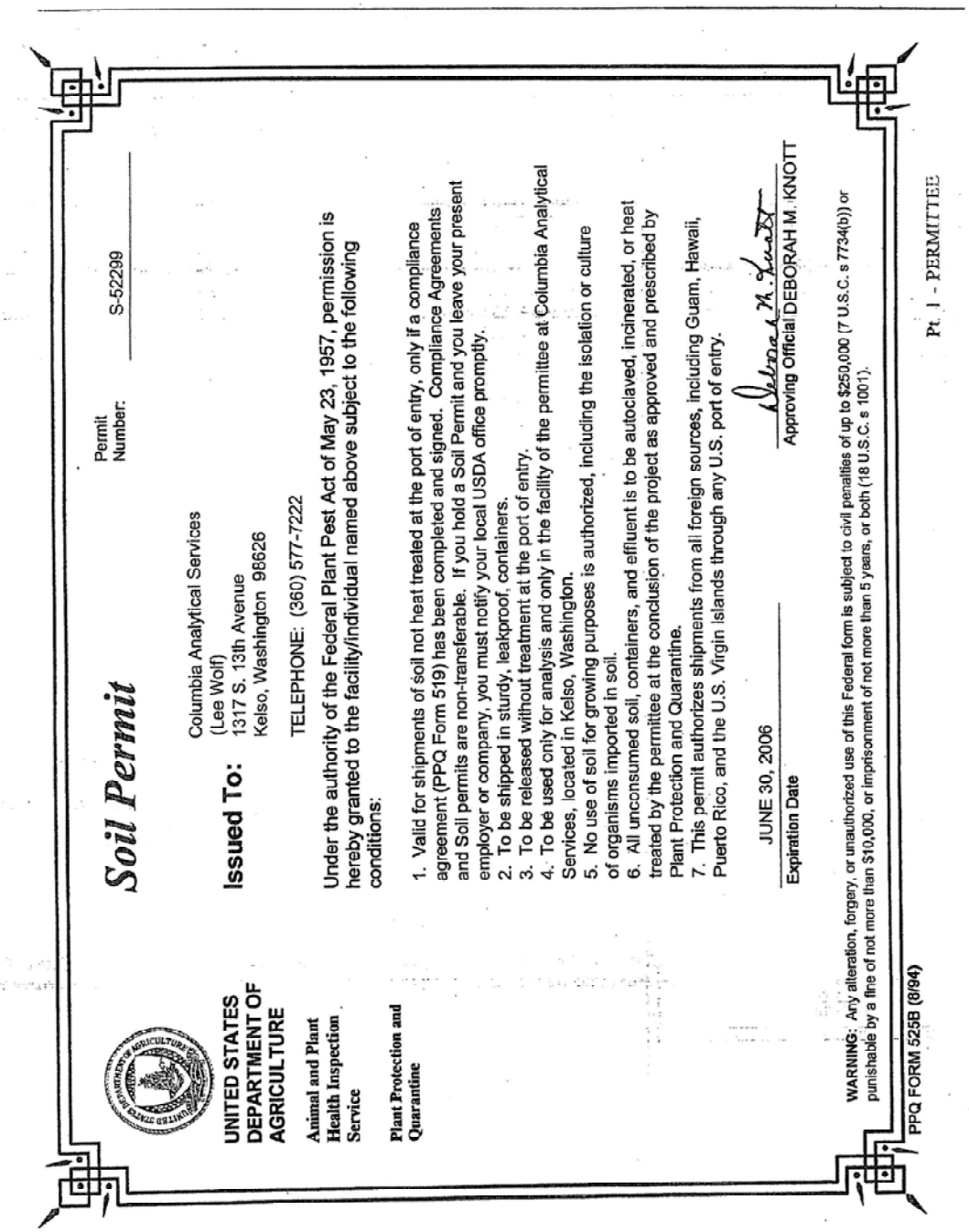
I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT

SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden _____ Joe Smith _____ 1/1/94 _____

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 8
Soil Import Permit



UNITED STATES DEPARTMENT OF AGRICULTURE

Animal and Plant Health Inspection Service

Plant Protection and Quarantine

Soil Permit

Permit Number: S-52299

Issued To: Columbia Analytical Services
(Lee Wolf)
1317 S. 13th Avenue
Kelso, Washington 98626

TELEPHONE: (360) 577-7222

Under the authority of the Federal Plant Pest Act of May 23, 1957, permission is hereby granted to the facility/individual named above subject to the following conditions:

1. Valid for shipments of soil not heat treated at the port of entry, only if a compliance agreement (PPQ Form 519) has been completed and signed. Compliance Agreements and Soil permits are non-transferable. If you hold a Soil Permit and you leave your present employer or company, you must notify your local USDA office promptly.
2. To be shipped in sturdy, leakproof, containers.
3. To be released without treatment at the port of entry.
4. To be used only for analysis and only in the facility of the permittee at Columbia Analytical Services, located in Kelso, Washington.
5. No use of soil for growing purposes is authorized, including the isolation or culture of organisms imported in soil.
6. All unconsumed soil, containers, and effluent is to be autoclaved, incinerated, or heat treated by the permittee at the conclusion of the project as approved and prescribed by Plant Protection and Quarantine.
7. This permit authorizes shipments from all foreign sources, including Guam, Hawaii, Puerto Rico, and the U.S. Virgin Islands through any U.S. port of entry.

JUNE 30, 2006
Expiration Date

Approving Official DEBORAH M. KNOTT

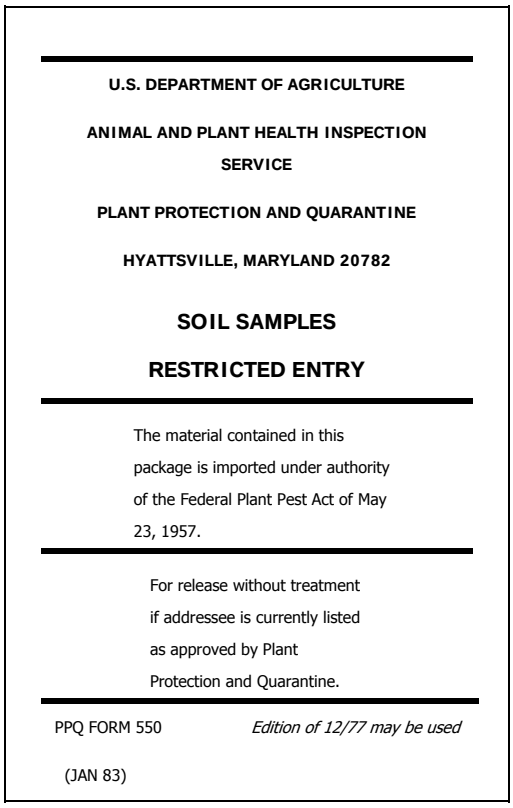
WARNING: Any alteration, forgery, or unauthorized use of this Federal form is subject to civil penalties of up to $250,000 (7 U.S.C. s 7734(b)) or punishable by a fine of not more than $10,000, or imprisonment of not more than 5 years, or both (18 U.S.C. s 1001).

PPQ FORM 525B (8/94)

Pt. 1 - PERMITTEE

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 9
Soil Samples Restricted Entry Labels



U.S. DEPARTMENT OF AGRICULTURE

ANIMAL AND PLANT HEALTH INSPECTION SERVICE

PLANT PROTECTION AND QUARANTINE

HYATTSVILLE, MARYLAND 20782

SOIL SAMPLES

RESTRICTED ENTRY

The material contained in this package is imported under authority of the Federal Plant Pest Act of May 23, 1957.

For release without treatment if addressee is currently listed as approved by Plant Protection and Quarantine.

PPQ FORM 550 *Edition of 12/77 may be used*

(JAN 83)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Investigation Derived Waste Management

Procedure 3-05

1.0 Purpose and Scope

This standard operating procedure (SOP) describes activities and responsibilities of the United States (U.S.) Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command, Atlantic (NAVFAC Atlantic) with regard to management of investigation-derived waste (IDW). The purpose of this procedure is to provide guidance for the minimization, handling, labelling, temporary storage, inventory, classification, and disposal of IDW generated under the ER Program. This procedure will also apply to personal protective equipment (PPE), sampling equipment, decontamination fluids, non-IDW trash, non-indigenous IDW, and hazardous waste generated during implementation of removal or remedial actions. The information presented will be used to prepare and implement work plans (WPs) for IDW-related field activities. The results from implementation of WPs will then be used to develop and implement final IDW disposal plans.

If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to IDW then those procedures may be added as an appendix to the project specific SAP.

This procedure applies to all Navy ER projects performed in the NAVFAC Atlantic Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by both the Contract Task Order (CTO) Manager and the Quality Assurance (QA) Manager or Technical Director, and documented.

This procedure was developed to serve as management-approved professional guidance for the management of IDW generated under the ER Program. It focuses on the requirements for minimizing, segregating, handling, labeling, storing, and inventorying IDW in the field. Certain drum inventory requirements related to the screening, sampling, classification, and disposal of IDW are also noted in this procedure.

2.0 Safety

The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the CTO WP and/or direction from the **Site Safety Officer (SSO)**.

All **Field Personnel** responsible for IDW management must adhere to the HASP and must wear the PPE specified in the site-specific HASP. Generally, this includes, at a minimum, steel-toed boots or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). If safe alternatives are not achievable, discontinue site activities immediately.

3.0 Terms and Definitions

None.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

4.0 Training and Qualifications

4.1 The **CTO Manager** is responsible for ensuring that IDW management activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all personnel involved in IDW management shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all IDW is managed according to this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

The equipment and supplies required for implementation of this SOP include the following:

- Containers for waste (e.g., [U.S. Department of Transportation] DOT approved 55-gallon open and closed top drums) and material to cover waste to protect from weather (e.g., plastic covering);
- Hazardous /non-hazardous waste drum labels (weatherproof);
- Permanent marking pens;
- Inventory forms for project file;
- Plastic garbage bags, zip lock storage bags, roll of plastic sheeting; and
- Steel-toed boots, chemical resistant gloves, coveralls, safety glasses, and any other PPE required in the HASP.

6.0 Procedure

The following procedures are used to handle the IDW.

6.1 Drum Handling

6.1.1 IDW shall be containerized using DOT approved drums. The drums shall be made of steel or plastic, have a 55-gallon capacity, be completely painted or opaque, and have removable lids (i.e., United Nations Code 1A2 or 1H2). Typically 55-gallon drums are used, however small drums may be used depending on the amount of waste generated. New steel drums are preferred over recycled drums.

6.1.2 Recycled drums should not be used for hazardous waste, PCBs or other regulated shipments. For short-term storage of liquid IDW prior to discharge, double-walled bulk steel or plastic storage tanks may be used. For this scenario, consider the scheduling and cost-effectiveness of this type of bulk storage, treatment, and discharge system versus longer-term drum storage.

6.1.3 For long-term IDW storage at other project locations, the DOT approved drums with removable lids are recommended. Verify the integrity of the foam or rubber sealing ring located on the underside of some drum lids prior to sealing drums containing IDW liquids.

6.1.4 If the ring is only partially attached to the drum lid, or if a portion of the ring is missing, select another drum lid with a sealing ring that is in sound condition.

6.1.5 To prepare IDW drums for labeling, wipe clean the outer wall surfaces and drum lids of all material that might prevent legible and permanent labeling. If potentially contaminated material adheres to the outer surface of a drum, wipe that material from the drum, and segregate the paper towel or rag used to remove the material with visibly soiled PPE and

disposable sampling equipment. Label all IDW drums and place them on pallets prior to storage.

6.2 Labelling

6.2.1 Containers used to store IDW must be properly labelled. Two general conditions exist: 1) from previous studies or on-site data, waste characteristics are known to be either hazardous or nonhazardous; or 2) waste characteristics are unknown until additional data are obtained.

6.2.2 For situations where the waste characteristics are known, the waste containers should be packaged and labelled in accordance with state regulations and any federal regulations that may govern the labelling of waste.

6.2.3 The following information shall be placed on all non-hazardous waste labels:

- Description of waste (i.e., purge water, soil cuttings);
- Contact information (i.e., contact name and telephone number);
- Date when the waste was first accumulated.

6.2.4 The following information shall be placed on all hazardous waste labels:

- Description of waste (i.e., purge water, soil cuttings);
- Generator information (i.e., name, address, contact telephone number);
- EPA identification number (supplied by on-site client representative);
- Date when the waste was first accumulated.

6.2.5 When the final characterization of a waste is unknown, a notification label should be placed on the drum with the words "waste characterization pending analysis" and the following information included on the label:

- Description of waste (i.e., purge water, soil cuttings);
- Contact information (i.e., contact name and telephone number);
- Date when the waste was first accumulated.

6.2.6 Once the waste has been characterized, the label should be changed as appropriate for a nonhazardous or hazardous waste.

6.2.7 Waste labels should be constructed of a weatherproof material and filled out with a permanent marker to prevent being washed off or becoming faded by sunlight. It is recommended that waste labels be placed on the side of the container, since the top is more subject to weathering. However, when multiple containers are accumulated together, it also may be helpful to include labels on the top of the containers to facilitate organization and disposal.

6.2.8 Each container of waste generated shall be recorded in the field notebook used by the person responsible for labelling the waste. After the waste is disposed of, either by transportation off-site or disposal on-site in an approved disposal area, an appropriate record shall be made in the same field notebook to document proper disposition of IDW.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

6.3 Types of Site Investigation Waste

Several types of waste are generated during site investigations that may require special handling. These include solid, liquid, and used PPE, as discussed further below.

Solid Waste

Soil cuttings from boreholes will typically be placed in containers unless site specific requirements allow for soil cuttings to be placed back into the borehole after drilling is complete. Drilling mud generated during investigation activities shall be collected in containers. Covers should be included on the containers and must be secured at all times and only open during filling activities. The containers shall be labelled in accordance with this SOP. An inventory containing the source, volume, and description of material put in the containers shall be logged on prescribed forms and kept in the project file.

Non-hazardous solid waste can be disposed on-site in the designated site landfill or in a designated evaporation pond if it is liquefied. Hazardous wastes must be disposed off-site at an approved hazardous waste landfill.

Liquid Waste

Groundwater generated during monitoring well development, purging, and sampling can be collected in truck-mounted containers and/or other transportable containers (i.e., 55-gallon drums). Lids or bungs on drums must be secured at all times and only open during filling or pumping activities. The containers shall be labelled in accordance with this SOP. Non-hazardous liquid waste can be disposed of in one of the designated lined evaporation ponds on-site. Hazardous wastes must be handled separately and disposed off-site at an approved hazardous waste facility.

Personal Protective Equipment

PPE that is generated throughout investigation activities shall be placed in plastic garbage bags. If the solid or liquid waste that was being handled is characterized as hazardous waste, then the corresponding PPE should also be disposed as hazardous waste. If not, all PPE should be disposed as non-hazardous waste in the designated on-site landfill. Trash that is generated as part of field activities may be disposed of in the landfill as long as the trash was not exposed to hazardous media.

6.4 Waste Accumulation On-Site

6.4.1 Solid, liquid, or PPE waste generated during investigation activities that are classified as nonhazardous or "characterization pending analysis" should be disposed of as soon as possible. Until disposal, such containers should be inventoried, stored as securely as possible, and inspected regularly, as a general good practice.

6.4.2 Solid, liquid, or PPE waste generated during investigation activities that are classified as hazardous shall not be accumulated on-site longer than 90 days. All hazardous waste containers shall be stored in a secured storage area. The following requirements for the hazardous waste storage area must be implemented:

- Proper hazardous waste signs shall be posted as required by any state or federal statutes that may govern the labelling of waste;
- Secondary containment to contain spills;
- Spill containment equipment must be available;
- Fire extinguisher;
- Adequate aisle space for unobstructed movement of personnel.

6.4.3 Weekly storage area inspections shall be performed and documented to ensure compliance with these requirements. Throughout the project, an inventory shall be maintained to itemize the type and quantity of the waste generated.

6.5 Waste Disposal

6.5.1 Solid, liquid, and PPE waste will be characterized for disposal through the use of client knowledge, laboratory analytical data created from soil or groundwater samples gathered during the field activities, and/or composite samples from individual containers.

6.5.2 All waste generated during field activities will be stored, transported, and disposed of according to applicable state, federal, and local regulations. All wastes classified as hazardous will be disposed of at a licensed treatment storage and disposal facility or managed in other approved manners.

6.5.3 In general, waste disposal should be carefully coordinated with the facility receiving the waste. Facilities receiving waste have specific requirements that vary even for non-hazardous waste, so characterization should be conducted to support both applicable regulations and facility requirements.

6.6 Regulatory Requirements

The following federal and state regulations shall be used as resources for determining waste characteristics and requirements for waste storage, transportation, and disposal:

- Code of Federal Regulations (CFR), Title 40, Part 261;
- CFR, Title 49, Parts 172, 173, 178, and 179.

6.7 Waste Transport

A state-certified hazardous waste hauler shall transport all wastes classified as hazardous. Typically, the facility receiving any waste can coordinate a hauler to transport the waste. Shipped hazardous waste shall be disposed of in accordance with all RCRA/USEPA requirements. All waste manifests or bills of lading will be signed either by the client or the client's designee.

7.0 Quality Control and Assurance

7.1 Management of IDW must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

8.1 Maintain records as required by implanting the procedures in this SOP.

8.2 Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual.* Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/-fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of Energy, United States (DOE). 1994. *The Off-Site Rule*. EH-231-020/0194. Office of Environmental Guidance. March.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

1999. *Management of Remediation Waste under the Resource Conservation and Recovery Act (RCRA)*. Office of Environmental Policy and Assistance. 20 December.

Department of the Navy (DON). 2001. *Department of the Navy Installation Restoration Manual. 2001 Update.* Draft. Alexandria, VA: Naval Facilities Engineering Command. August.

2007. *Navy Environmental and Natural Resources Program Manual.* OPNAV Instruction 5090.1c . October.

Environmental Protection Agency, United States (EPA). 1991. *Management of Investigative-Derived Wastes During Site Inspections*. Office of Emergency and Remedial Response. EPA/540/G-91/009. May.

1992a. *Guidance for Performing Site Inspections under CERCLA*. EPA/540/R-92/021. Office of Emergency and Remedial Response. September.

1992b. *Guide to Management of Investigative-Derived Wastes*. Quick reference fact sheet. OSWER Dir. 9345.3-03FS. Office of Solid Waste and Emergency Response. January.

1997a. *Sending Wastes Off Site? OSC and RPM Responsibilities under the Off-Site Rule*. EPA/540-F-97-006, Office of Solid Waste and Emergency Response. September.

1997b. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Final Update IIIA. Office of Solid Waste. Updates available: www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

1998. *Management of Remediation Waste under RCRA*. EPA/530-F-98-026. Office of Solid Waste and Emergency Response. October.

(No Date). *Compliance with the Off-Site Rule During Removal Actions.* Office of Regional Counsel (Region 3). Hendershot, Michael.

NAVFAC NW Standard Operating Procedure Number I-D-1, *Drum Sampling.*

NAVFAC NW Standard Operating Procedure Number I-F, *Equipment Decontamination.*

NAVFAC NW Standard Operating Procedure Number III-D, *Logbooks.*

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

Equipment Decontamination

Procedure 3-06

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes methods of equipment decontamination, to be used for activities where samples for chemical analysis are collected or where equipment will need to be cleaned before leaving the site or before use in subsequent activities.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

It is the responsibility of the **Site Safety Officer (SSO)** to set up the site zones (i.e., exclusion, transition, and clean) and decontamination areas. Generally the decontamination area is located within the transition zone, upwind of intrusive activities, and serves as the washing area for both personnel and equipment to minimize the spread of contamination into the clean zone. Typically, for equipment, a series of buckets are set up on a visqueen-lined bermed area. Separate spray bottles containing cleaning solvents as described in this procedure or the Contract Task Order (CTO) Work Plan (WP) and distilled water are used for final rinsing of equipment. Depending on the nature of the hazards and the site location, decontamination of heavy equipment, such as augers, pump drop pipe, and vehicles, may be accomplished using a variety of techniques.

All **Field Personnel** responsible for equipment decontamination must adhere to the site-specific health and safety plan (HSP) and must wear the personal protective equipment (PPE) specified in the site-specific HSP. Generally this includes, at a minimum, Tyvek® coveralls, steel-toed boots with boot covers or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). Air monitoring by the **SSO** may result in an upgrade to the use of respirators and cartridges in the decontamination area; therefore, this equipment must be available on site. If safe alternatives are not achievable, discontinue site activities immediately.

In addition to the aforementioned precautions, the following sections describe safe work practices that will be employed.

2.1 **Chemical Hazards associated with Equipment Decontamination**

- Avoid skin contact with and/or incidental ingestion of decontamination solutions and water.
- Utilize PPE as specified in the site-specific HSP to maximize splash protection.
- Refer to material safety data sheets, safety personnel, and/or consult sampling personnel regarding appropriate safety measures (i.e., handling, PPE including skin and respiratory).
- Take the necessary precautions when handling detergents and reagents.

2.2 **Physical Hazards associated with Equipment Decontamination**

- To avoid possible back strain, it is recommended to raise the decontamination area 1 to 2 feet above ground level.
- To avoid heat stress, over exertion, and exhaustion, it is recommended to rotate equipment decontamination among all site personnel.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- Take necessary precautions when handling field sampling equipment.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **CTO Manager** is responsible for ensuring that decontamination activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all personnel involved in equipment decontamination shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all field equipment is decontaminated according to this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Procedure

Decontamination of equipment used in soil/sediment sampling, groundwater monitoring, well drilling and well development, as well as equipment used to sample groundwater, surface water, sediment, waste, wipe, asbestos, and unsaturated zone, is necessary to prevent cross-contamination and to maintain the highest integrity possible in collected samples. Planning a decontamination program requires consideration of the following factors:

- Location where the decontamination procedures will be conducted
- Types of equipment requiring decontamination
- Frequency of equipment decontamination
- Cleaning technique and types of cleaning solutions appropriate to the contaminants of concern
- Method for containing the residual contaminants and wash water from the decontamination process
- Use of a quality control measure to determine the effectiveness of the decontamination procedure

The following subsections describe standards for decontamination, including the frequency of decontamination, cleaning solutions and techniques, containment of residual contaminants and cleaning solutions, and effectiveness.

5.1 Decontamination Area

Select an appropriate location for the decontamination area at a site based on the ability to control access to the area, the ability to control residual material removed from equipment, the need to store clean equipment, and the ability to restrict access to the area being investigated. Locate the decontamination area an adequate distance away and upwind from potential contaminant sources to avoid contamination of clean equipment.

5.2 Types of Equipment

Drilling equipment that must be decontaminated includes drill bits, auger sections, drill-string tools, drill rods, split barrel samplers, tremie pipes, clamps, hand tools, and steel cable. Decontamination of monitoring well development and groundwater sampling equipment includes submersible pumps, bailers, interface probes, water level meters, bladder pumps, airlift pumps, peristaltic pumps, and lysimeters. Other sampling equipment that requires decontamination includes, but is not limited to, hand trowels,

hand augers, slide hammer samplers, shovels, stainless-steel spoons and bowls, soil sample liners and caps, wipe sampling templates, composite liquid waste samplers, and dippers. Equipment with a porous surface, such as rope, cloth hoses, and wooden blocks, cannot be thoroughly decontaminated and shall be properly disposed of after one use.

5.3 Frequency of Equipment Decontamination

Decontaminate down-hole drilling equipment and equipment used in monitoring well development and purging prior to initial use and between each borehole or well. Down-hole drilling equipment, however, may require more frequent cleaning to prevent cross-contamination between vertical zones within a single borehole. When drilling through a shallow contaminated zone and installing a surface casing to seal off the contaminated zone, decontaminate the drilling tools prior to drilling deeper. Initiate groundwater sampling by sampling groundwater from the monitoring well where the least contamination is suspected. Decontaminate groundwater, surface water, and soil sampling devices prior to initial use and between collection of each sample to prevent the possible introduction of contaminants into successive samples.

5.4 Cleaning Solutions and Techniques

Decontamination can be accomplished using a variety of techniques and fluids. The preferred method of decontaminating major equipment, such as drill bits, augers, drill string, and pump drop-pipe, is steam cleaning. To steam clean, use a portable, high-pressure steam cleaner equipped with a pressure hose and fittings. For this method, thoroughly steam wash equipment and rinse it with potable tap water to remove particulates and contaminants.

A rinse decontamination procedure is acceptable for equipment such as bailers, water level meters, new and re-used soil sample liners, and hand tools. The decontamination procedure shall consist of the following: (1) wash with a non-phosphate detergent (Alconox®, Liquinox®, or other suitable detergent) and potable water solution; (2) rinse with potable water; (3) spray with laboratory-grade isopropyl alcohol; (4) rinse with deionized or distilled water; and (5) spray with deionized or distilled water. If possible, disassemble equipment prior to cleaning. Add a second wash at the beginning of the process if equipment is very soiled.

Decontaminating submersible pumps requires additional effort because internal surfaces become contaminated during usage. Decontaminate these pumps by washing and rinsing the outside surfaces using the procedure described for small equipment or by steam cleaning. Decontaminate the internal surfaces by recirculating fluids through the pump while it is operating. This recirculation may be done using a relatively long (typically 4 feet) large-diameter pipe (4-inch or greater) equipped with a bottom cap. Fill the pipe with the decontamination fluids, place the pump within the capped pipe, and operate the pump while recirculating the fluids back into the pipe. The decontamination sequence shall include: (1) detergent and potable water; (2) potable water rinse; (3) potable water rinse; and (4) deionized water rinse. Change the decontamination fluids after each decontamination cycle.

Solvents other than isopropyl alcohol may be used, depending upon the contaminants involved. For example, if polychlorinated biphenyls or chlorinated pesticides are contaminants of concern, hexane may be used as the decontamination solvent; however, if samples are also to be analyzed for volatile organics, hexane shall not be used. In addition, some decontamination solvents have health effects that must be considered. Decontamination water shall consist of distilled or deionized water. Steam-distilled water shall not be used in the decontamination process as this type of water usually contains elevated concentrations of metals. Decontamination solvents to be used during field activities will be specified in the CTO WP.

Rinse equipment used for measuring field parameters, such as pH (indicates the hydrogen ion concentration – acidity or basicity), temperature, specific conductivity, and turbidity with deionized or distilled water after each measurement. Also wash new, unused soil sample liners and caps with a fresh

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

detergent solution and rinse them with potable water followed by distilled or deionized water to remove any dirt or cutting oils that might be on them prior to use.

5.5 **Containment of Residual Contaminants and Cleaning Solutions**

A decontamination program for equipment exposed to potentially hazardous materials requires a provision for catchment and disposal of the contaminated material, cleaning solution, and wash water.

When contaminated material and cleaning fluids must be contained from heavy equipment, such as drill rigs and support vehicles, the area must be properly floored, preferably with a concrete pad that slopes toward a sump pit. If a concrete pad is impractical, planking can be used to construct solid flooring that is then covered by a nonporous surface and sloped toward a collection sump. If the decontamination area lacks a collection sump, use plastic sheeting and blocks or other objects to create a bermed area for collection of equipment decontamination water. Situate items, such as auger flights, which can be placed on metal stands or other similar equipment, on this equipment during decontamination to prevent contact with fluids generated by previous equipment decontamination. Store clean equipment in a separate location to prevent recontamination. Collect decontamination fluids contained within the bermed area and store them in secured containers as described below.

Use wash buckets or tubs to catch fluids from the decontamination of lighter-weight drilling equipment and hand-held sampling devices. Collect the decontamination fluids and store them on site in secured containers, such as U.S. Department of Transportation-approved drums, until their disposition is determined by laboratory analytical results. Label containers in accordance with Procedure 3-05, *IDW Management*.

6.0 Quality Control and Assurance

A decontamination program must incorporate quality control measures to determine the effectiveness of cleaning methods. Quality control measures typically include collection of equipment blank samples or wipe testing. Equipment blanks consist of analyte-free water that has been poured over or through the sample collection equipment after its final decontamination rinse. Wipe testing is performed by wiping a cloth over the surface of the equipment after cleaning. These quality control measures provide "after-the fact" information that may be useful in determining whether or not cleaning methods were effective in removing the contaminants of concern.

7.0 Records, Data Analysis, Calculations

Any project where sampling and analysis is performed shall be executed in accordance with an approved sampling and analysis plan. This procedure may be incorporated by reference or may be incorporated with modifications described in the plan.

Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or References

8.1 ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites.* ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. www.astm.org.

8.2 NAVSEA T0300-AZ-PRO-010. *Navy Environmental Compliance Sampling and Field Testing Procedures Manual*. August 2009.

8.3 Procedure 3-05, *IDW Management*.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Land Surveying

Procedure 3-07

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) for acquiring land surveying data to facilitate the location and mapping of geologic, hydrologic, geotechnical data, and analytical sampling points and to establish topographic control over project sites.

1.2 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific SAP.

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

1.5 If there are procedures, whether it be from Resolution Consultants, state and/or federal, that are not addressed in this SOP and are applicable to land surveying then those procedures may be added as an appendix to the project specific Sampling and Analysis Plan (SAP).

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to conducting fieldwork. All **field sampling personnel** must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the specific field tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety Officer (SSO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the HASP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSO.

2.4 The health and safety considerations for the work associated with land surveying include:

- Slip, trips and falls associated with work in the field;

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- Biological hazards associated with work in the field; and,
- Potential hazards associated with contaminants of concern (COC) that may be located in the survey area,

3.0 Terms and Definitions

3.1 Boundary Survey

Boundary surveys are conducted by Certified Land Surveyors in order to delineate a legal property line for a site or section of a site.

3.2 Global Positioning System (GPS)

A system of satellites, computers, and receivers that is able to determine the latitude and longitude of a receiver on Earth by calculating the time difference for signals from different satellites to reach the receiver.

4.0 Interferences

4.1 Commercially available GPS units typically have a level of precision of (±) 3 to 5 meters. Field corrections can be made as described in Section 8.3 below.

5.0 Training and Qualifications

5.1 Qualifications and Training

5.1.1 The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **Contract Task Order (CTO) Manager** is responsible for ensuring that land surveying activities comply with this procedure. The CTO Manager is responsible for ensuring that all field sampling personnel involved in land surveying shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager (FM)** is responsible for ensuring that all field personnel follow these procedures. In virtually all cases, subcontractors will conduct these procedures. The FM or designee is responsible for overseeing the activities of the subcontractor and ensuring that sampling points and topographic features are properly surveyed.

6.0 Equipment and Supplies

6.1 The following equipment list contains materials that may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Personal protective equipment (PPE) and other safety equipment, as required by the HASP;
- Commercially available GPS unit; and,
- Field Logbook.

7.0 Calibration or Standardization

7.1 An authorized manufacturer's representative shall inspect and calibrate survey instruments in accordance with the manufacturer's specifications regarding procedures and frequencies. At a minimum, instruments shall be calibrated no more than six months prior to the start of the survey work.

7.2 Standards for all survey work shall be in accordance with National Oceanic and Atmospheric Administration standards and, at a minimum, with accuracy standards set forth below. The horizontal accuracy for the location of all grid intersection and planimetric features shall be (±) 0.1 feet. The horizontal accuracy for boundary surveys shall be 1 in 10,000 feet (1:10,000). The vertical accuracy for ground surface elevations shall be (±) 0.1 feet. Benchmark elevation accuracy and elevation of other permanent features, including monitoring wellheads, shall be (±) 0.01 feet.

8.0 Procedure

8.1 Theodolite/Electronic Distance Measurement (EDM)

Follow the procedures listed below during theodolite/EDM land surveying conducted under the NAVFAC CLEAN Program:

- A land surveyor registered in the state or territory in which the work is being performed shall directly supervise all surveying work.
- Reference surveys to the local established coordinate systems and base all elevations and benchmarks established on U.S. Geological Survey datum, 1929 general adjustment.
- Reference surveyed points to Mean Sea Level (Lower Low Water Level).
- Jointly determine appropriate horizontal and vertical control points prior to the start of survey activities. If discrepancies in the survey (e.g., anomalous water level elevations) are observed, the surveyor may be required to verify the survey by comparison to a known survey mark. If necessary, a verification survey may be conducted by a qualified third party.
- All field notes, sketches, and drawings shall clearly identify the horizontal and vertical control points by number designation, description, coordinates, and elevations. Map all surveyed locations using a base map or other site mapping, as specified by the project Work Plan or SAP.
- Begin and end all surveys at the designated horizontal and vertical control points to determine the degree of accuracy of the surveys.
- Iron pins used to mark control points shall be made of reinforcement steel or an equivalent material and shall be 18 inches long with a minimum diameter of 5/8 inch. Drive pins to a depth of 18 inches into the soil.
- Stakes used to mark survey lines and points shall be made from 3-foot lengths of 2-inch by 2-inch lumber and pointed at one end. Clearly mark them with brightly colored weatherproof flagging and paint.
- Clearly mark the point on a monitoring well casing or well riser that is surveyed by filing grooves into the casing/riser on either side of the surveyed point, or by marking the riser with a permanent ink marker.

8.2 Global Positioning System (GPS) to Conduct Land Survey

Follow the procedures listed below during land surveying using GPS:

- A land surveyor registered in the state or territory in which the work is being performed shall directly supervise all surveying work.
- Reference surveys to the local established coordinate systems and base all elevations and benchmarks established on U.S. Geological Survey datum, 1929 general adjustment.

- All field notes, sketches, and drawings shall clearly identify the horizontal and vertical control points by number designation, description, coordinates, and elevations. Map all surveyed locations using a base map or other site mapping, as specified in the project Work Plan or SAP.
- Begin and end all surveys at the designated horizontal and vertical control points (as applicable) to determine the degree of accuracy of the surveys.
- Iron pins used to mark control points shall be made of reinforcement steel or an equivalent material and shall be 18 inches long with a minimum diameter of 5/8 inch. Drive pins to a depth of 18 inches into the soil.
- Stakes used to mark survey lines and points shall be made from 3-foot lengths of 2-inch by 2-inch lumber and pointed at one end. Clearly mark them with brightly colored weatherproof flagging and paint.
- Clearly mark the point on a monitoring well casing that is surveyed by filing grooves into the casing on either side of the surveyed point.

8.3 Global Positioning System (GPS) to Position Sample Locations or Locate Site Features

Experienced field personnel may use a GPS system unit to position sample locations (e.g. grid positioned samples, soil boring locations) at a site. The decision to use field personnel or a licensed land surveyor will depend on the objectives of the survey (e.g. vertical elevation is not required) and the levels of precision required. Typically when a level of precision greater than (±) 3 to 5 meters is required, a licensed surveyor will be required. When a level of precision of (±) 3 to 5 meters is sufficient to meet project requirements (i.e. when laying sampling grids, identifying significant site features, or locating features identified in GIS figures) experienced field personnel may use commercially available, consumer-grade GPS units. Follow the procedures listed below to locate samples or site features using GPS:

- A commercially available GPS unit with Wide Angle Averaging System (WAAS), topographic map display, and waypoint storage capabilities should be used.
- If waypoints are to be imported into a GIS database, the same grid projection system should be used.
- If a permanent reference point near the site is available, it is recommended that a waypoint at this location be taken every day waypoints are stored.
- When laying out a sampling grid from a GIS map, upload the coordinates from GIS to the GPS unit, including coordinates for an easily identified, permanent, nearby feature (i.e. building corner, roadway intersection, or USGS benchmark).
- If during the initial site walk, the permanent feature identified does not overlay within (±) 5 meters as identified in the GPS unit, field corrections of the waypoints should be made.
- Field corrections can be made by adding/subtracting the difference in x,y coordinates between the field measurement of the permanent site feature and the anticipated x,y coordinates. This correction should then be applied to the x,y coordinates for each sampling location to be marked. Corrected x,y coordinates can then be uploaded into the GPS unit.
- Sampling points and site features can then be located in the field using the GPS units “Go To” function. When the distance to the sampling point or feature remains close to zero, the location can be marked.
- If no field corrections to the sampling location need to be made, or if sampling locations are to be surveyed by a licensed surveyor at a later date, no additional waypoints need to be taken. If significant changes to the sampling location are made, GPS coordinates at the corrected location shall be stored and labeled.

- It is recommended that GPS coordinates be uploaded to a storage device such as PC at the end of each day.
- Field logs shall indicate manufacturer and model number for GPS unit used, map datum and projection used, and any field corrections made. If the GPS unit cannot lock onto a WAAS system at the site, this should also be noted.

9.0 Quality Control and Assurance

None.

10.0 Data and Records Management

The surveyor shall record field notes daily using generally accepted practices. The data shall be neat, legible, in indelible ink, and easily reproducible. Copies of the surveyor's field notes and calculation forms generated during the work shall be obtained and placed in the project files.

Surveyor's field notes shall, at a minimum, clearly indicate:

- The date of the survey;
- General weather conditions;
- The name of the surveying firm;
- The names and job titles of personnel performing the survey work;
- Equipment used, including serial numbers; and,
- Field book designations, including page numbers.

A land surveyor registered in the state or territory in which the work was done shall sign, seal, and certify the drawings and calculations submitted by the surveyor.

Dated records of land surveying equipment calibration shall be provided by the surveyor and placed in the project files. Equipment serial numbers shall be provided in the calibration records.

11.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Monitoring Well Installation

Procedure 3-12

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the methods to be used during the installation of groundwater monitoring wells. It describes the components of monitoring well design and installation and sets forth the rationale for use of various well installation techniques in specific situations.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**.

2.2 Before well installation commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated well locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.

2.3 Physical Hazards Associated with Well Installation

- Stay clear of all moving equipment and avoid wearing loose fitting clothing.
- When using an approved retractable-blade knife, always cut away from one self and make sure there are no other people in the cutting path or the retractable-blade knife.
- To avoid slip/trip/fall conditions during drilling activities, keep the area clear of excess soil cuttings and groundwater. Use textured boots/boot cover bottoms in muddy areas.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and personal protective equipment (PPE), drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
- To avoid hazards associated with subsurface utilities, ensure all sampling locations have been properly surveyed as described in SOP 3-01, Utility Clearance.
- Be aware of restricted mobility caused by PPE.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

3.0 Terms and Definitions

3.1 **Annulus:** The annulus is the down-hole space between the borehole wall and the well casing and screen.

3.2 **Bridge:** A bridge is an obstruction in the drill hole or annulus. A bridge is usually formed by caving of the wall of the well bore, by the intrusion of a large boulder, or by the placement of filter pack materials during well completion. Bridging can also occur in the formation during well development.

3.3 **Filter Pack:** Filter pack is sand or gravel that is smooth, uniform, clean, well-rounded, and siliceous. It is placed in the annulus of the well between the borehole wall and the well screen to prevent formation materials from entering the well and to stabilize the adjacent formation.

3.4 **Grout:** Grout is a fluid mixture of cement and water that can be forced through a tremie pipe and emplaced in the annular space between the borehole and casing to form an impermeable seal. Various additives, such as sand, bentonite, and polymers, may be included in the mixture to meet certain requirements.

3.5 **Heaving (Running) Sands:** Loose sands in a confined water-bearing zone or aquifer which tend to rise up into the drill stem when the confining unit is breached by the drill bit. Heaving sands occur when the water in the aquifer has a pressure head great enough to cause upward flow into the drill stem with enough velocity to overcome the weight of the sand.

3.6 **Sieve Analysis:** Sieve analysis is the evaluation of the particle-size distribution of a soil, sediment, or rock by measuring the percentage of the particles that will pass through standard sieves of various sizes.

4.0 Interferences

4.1 Heaving sands may be problematic in unconsolidated sands encountered below the water table.

4.2 Rotary drilling methods requiring bentonite-based drilling fluids should be used with caution to drill boreholes that will be used for monitoring well installation. The bentonite mud builds up on the borehole walls as a filter cake and permeates the adjacent formation, potentially reducing the permeability of the material adjacent to the well screen.

4.3 If water or other drilling fluids have been introduced into the boring during drilling or well installation, samples of these fluids should be obtained and analyzed for chemical constituents that may be of interest at the site. In addition, an attempt should be made to recover the quantity of fluid or water that was introduced, either by flushing the borehole prior to well installation and/or by overpumping the well during development.

4.4 Track-mounted drill rigs are suitable for travelling on many types of landscapes that truck-mounted units cannot access, but may have limitations on extremely uneven or soft terrain.

4.5 Care should be taken to prevent cross-contamination between well locations. All drilling equipment coming in contact with potentially contaminated soil and/or groundwater will be decontaminated by the drilling subcontractor prior to initial drilling activities and between drilling locations in accordance with SOP 3-06, Equipment Decontamination.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

5.2 **Responsibilities**

5.2.1 **Contract Task Order (CTO) Managers** are responsible for issuing sampling and analysis plans (SAPs) that reflect the procedures and specifications presented in this procedure. Individual municipalities, county agencies, and possibly state regulatory agencies enforce regulations that may include well construction and installation requirements. The **CTO Manager** shall be familiar with current local and state regulations, and ensure that these regulations are followed. The **CTO Manager** is responsible for ensuring that all personnel involved in monitoring well installation shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager** is responsible for direct supervision of the installation of monitoring wells and ensuring that procedures and specifications are implemented in the field in accordance with the approved SAP and well installation permits. The qualifications for the **Field Manager** must be in accordance with local jurisdictions with authority over the operations conducted.

5.2.4 All field personnel are responsible for the implementation of this procedure.

5.2.5 The on-site hydrogeologist/engineer is expected to obtain a description of the lithologic samples obtained during the excavation and construction of a monitoring well. These data are often required to provide guidance regarding the installation of specific components of the monitoring well. Guidance for lithologic sample collection and sample description is contained within SOP 3-16, Soil and Rock Classification.

6.0 Equipment and Supplies

6.1 Materials provided by the drilling contractor may include:

- Drill rig, drill rods, hollow stem augers, etc.
- Decontamination equipment (e.g., steam cleaner, high-pressure washer, brushes, etc.)
- Decontamination pad materials
- Well screen/riser pipe with flush-threaded couplings including riser and bottom caps
- Clean, filter sand
- Bentonite chips or pellets
- Cement grout and tremie pipe
- Portland cement for well pad completion
- Steel protective riser covers and locking caps
- Weighted calibrated tape
- Split-spoon samplers
- 55-gallon drums or containers for drill cuttings, decontamination fluids, etc.

6.2 In addition to those materials provided by the drilling contractor, equipment and materials required by the project geologist/engineer may include, but is not limited to, the following:

- Photoionization Detector (PID)
- Spill kit, including at a minimum sorbent pads and shovel (if not provided by subcontractor)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- Plastic sheeting
- Teaspoon or spatula
- Resealable plastic bags
- Boring Log Records
- Decontamination materials (per SOP No. 3-06 - Equipment Decontamination)
- Weighted measuring tape for depth measurement
- Soil logging materials (e.g. USCS classification field card, millimeter rule, hand lens, etc.)
- Survey lathes or pin flags
- Digital camera
- PPE as required by the HASP
- Planning documents including the site-specific HASP and SAP
- Large indelible ink or paint pen
- Field logbook/field forms/site maps (water proof)

7.0 Procedure

7.1 General Procedures

- Specific drilling, sampling, and installation equipment and methodology will be dictated by the type of well to be installed (e.g., single case (Type II), double case (Type III), bedrock, etc.), geologic characteristics of the site, the type of contaminants being monitored, and local and state regulations.
- For access to locations when travelling over difficult terrain, an appropriate line should be chosen before mobilizing the drill rig or other support vehicles. If clearing of trees or ground cover is required, perform these activities in advance to avoid down time. Avoid wet or soft areas where possible or use ground mats and/or timbers to aid in supporting the rig as it travels. If drilling on soft material, place geomatting and ground mats under the rig tracks or stabilizers prior to drilling.
- A utility locate must be conducted to identify all underground utilities at the site prior to drilling (refer to SOP 3-01, Utility Clearance*)*. Proper clearance procedures for aboveground/overhead utilities must also be followed as specified in the HASP.
- Although new well materials (well screen and riser pipe) generally arrive at the site boxed and sealed within plastic bags, it is sometimes necessary to decontaminate the materials prior to their use. Well materials should be inspected by the project geologist/engineer upon delivery to check for cleanliness. If the well materials appear dirty, or if local or regional regulatory guidance requires decontamination, then well material decontamination should be performed by the drilling subcontractor in accordance with SOP 3-06, Equipment Decontamination.
- The diameter of the borehole must be a minimum of 2 inches greater than the outside diameter of the well screen or riser pipe used to construct the well. This is necessary so that sufficient annular space is available to install filter packs, bentonite seals, and grout seals, and allow the passage of tremie pipe where grouting at depth is required. Bedrock wells may require reaming after coring in order to provide a large enough borehole diameter for well installation.
- When soil sampling is required (refer to the SAP), soil samples will be collected for visual logging by advancing split-spoon samplers through the augers. The soil will be visually logged by a field geologist and include lithologic characteristics (i.e., soil type, color, density, moisture content, etc.) using the the

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

methods described in SOP 3-16, Soil and Rock Classification. This information will be recorded on a boring/well log form, along with well construction details.

7.2 Drilling Techniques

Drilling of monitoring well boreholes may be accomplished by a variety of methods as described below. Preferred methods include those that temporarily case the borehole during drilling (i.e., hollow stem auger and sonic methods) using an override system. Other methods can be used where specific subsurface conditions or well design criteria dictate.

- Hollow stem auger (HSA) – Borings are advanced by rotating steel hollow stem augers with an attached cutting head. Soil cuttings are displaced by the cutting head and transported to the surface via continuous spiral flights attached to each auger stem. This method is widely used for unconsolidated soils that have a tendency to collapse within the boring. A bottom plug can be placed in the bottom auger to prevent soils from entering and clogging the auger, especially in the case of heaving sands. However, a bottom plug cannot be used when soil samples are to be collected through the augers. Soil plugs that accumulate in the bottom of the auger must be removed or knocked out prior to sampling or well installation.
- Solid stem auger – This type of drilling method is similar to HSA drilling using a solid stem or sealed hollow stem auger flights to advance the boring. Solid stem, continuous flight auger use is limited to semi-consolidated sediments or to cohesive or semi-cohesive unconsolidated sediments that don't have a tendency to collapse when disturbed.
- Sonic methods – Sonic drilling consists of advancing concentric hollow drill casings (inner and outer) using rotation in conjunction with axial vibration of the drill casing. Once the casings are advanced to the appropriate depth, the inner string is removed with a core of drill cuttings while the outer casing remains in place to keep the borehole open. Cuttings are removed from the inner casing relatively intact for logging or sampling purposes. This drilling method is used for a variety of soil types, from heaving sands to consolidated or indurated formations. Smearing of the formation along the borehole walls is minimal since moderate vibration and rotation techniques are used to advance the casings. Since the total borehole diameter in sonic drilling is only incrementally larger than the inner casing diameter, care should be taken during installation of the monitoring well to ensure the well is centered and adequate space is available for annular materials.
- Rotary methods (water or mud) – Rotary drilling methods consist of drill rods coupled to a drill bit that rotates and cuts through the soils to advance the borehole. Water or drilling fluid ("mud") is forced through the hollow drill rods and drill bit as the rods are rotated. The soil cuttings are forced up the borehole with the drilling fluids to the surface and the fluids recirculated. The drilling fluid provides a hydrostatic pressure that reduces or prevents the borehole from collapsing. Clean, potable water must be used for water-rotary drilling to prevent introducing trace contaminants. A sample of the potable water should be collected during the course of well installation for analysis of the same parameters defined for the groundwater samples. If mud-rotary is used to advance boreholes, potable water and bentonite drilling mud should only be used. No chemical additives shall be mixed in the drilling fluid to alter viscosity or lubricating properties. Adequate well development is essential for removal of drilling mud and fluids from the formation materials and ensure collection of representative groundwater samples.
- Rotary methods (Air) – Air rotary methods are similar to water rotary but use high air velocities in place of drilling fluids to rotate the drill bit and carry the soil cuttings up the borehole to the surface. Care must be taken to ensure that contaminants are not introduced into the air stream from compressor oils, etc. Most compressor systems are compatible with a coalescing filter system. Cuttings exiting the borehole under pressure must be controlled, especially when drilling in a zone of potential contamination. This can be accomplished by using an air diverter with hose or pipe to carry the cuttings to a waste container. Letting the cuttings blow uncontrolled from the borehole is not acceptable.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

7.3 Well Construction and Installation

- If rotary drilling techniques are used, the borehole should be flushed or blown free of material prior to well installation. If hollow stem augers are used, the soil or bottom plug should be removed and the augers raised approximately six inches above the bottom of the borehole, while slowly rotating the augers to remove cuttings from the bottom of the boring. The depth of the borehole should be confirmed with a weighted, calibrated tape.
- The riser pipe and screen should be connected with flush-threaded joints and assembled wearing clean, disposable gloves. No solvent or anti-seize compound should be used on the connections. The full length of the slotted portion of the well screen and unslotted riser pipe should be measured and these measurements recorded on a well construction form (Attachment 1).
- If placed in an open borehole, the assembled well should be carefully lowered and centered in the borehole so that the well is true, straight, and vertical throughout. Centering can also be accomplished with the use of centralizers, if necessary. However, centralizers should be placed so that they do not inhibit the installation of filter sand, bentonite seal, and annular grout. Wells less than 50 deep generally do not require centralizers.
- If hollow stem augers are used, the well should be lowered through the augers and each auger flight removed incrementally as the filter sand, bentonite seal, and grout are tremmied or poured into the annular space of the well. The well should be temporarily capped before filter sand and other annular materials are installed.
- Clean, silica sand should be placed around the well screen to at least 1 foot above the top of the screen. The filter sand should be appropriately graded and compatible with the selected screen size and surrounding formation materials. In general, the filter pack should not extend more than 3 feet above the top of the screen to limit the thickness of the monitoring zone. As the filter pack is placed, a weighted tape should be lowered in the annular space to verify the depth to the top of the layer. This measurement will be recorded on the well construction form (Attachment 1). If necessary, to eliminate possible bridging or creation of voids, placement of the sand pack may require the use of a tremie pipe. Tremie pipe sandpack installations are generally suggested for deeper wells and for wells which are screened some distance beneath the water table.
- A minimum 2-foot thick layer of bentonite pellets or slurry seal will be installed immediately above the filter sand to prevent vertical flow within the boring from affecting the screened interval. Bentonite chips/pellets must be hydrated if place above the water table prior to grouting. If bridging is of concern as in the case of deep wells, powdered bentonite may be mixed with water into a very thick slurry and a tremie pipe used to place the seal to the desired depth. Placement of the bentonite seal in the borehole will be recorded on the well construction form (Attachment 1).
- The remaining annular space around the well will be grouted from the top of the bentonite seal to the surface with a grout composed of neat cement, a bentonite cement mixture, or high solids sodium bentonite grout.
- Each well riser will be secured with an expandable, locking cap (vented if possible). Optionally, a hole can be drilled in the upper portion of the riser to allow venting of the well.
- The well will be completed within a concrete well pad consisting of a Portland cement/sand mixture. Well pads are generally 3 feet by 3 feet square but may be larger or smaller depending on site conditions and state-specific well construction standards. Round concrete well pads are also acceptable. A minimum of 1 inch of the finished pad should be below grade to prevent washing and undermining by soil erosion.
- If completed as a flush-mount well, the well riser will be cut off approximately 4 to 6 inches below ground surface and an expandable, locking cap placed on the well riser. The area around the riser is dug out and a steel well vault or manhole cover placed over the riser and set almost flush to the ground

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

to protect the well. The manhole cover should be water-tight and secured with bolts to prevent casual access. The well pad will then be constructed around the well vault and slightly mounded at the center and sloping away to prevent surface water from accumulating in the well vault.

- If completed as a stick-up well, the well riser is cut approximately 2.5 to 3 feet above the ground surface and an expandable, locking cap placed on the well riser. A steel guard pipe with hinged, locking cap is placed over the well riser as a protective casing. The bottom of the guard pipe will be set approximately 2 feet below ground surface and sealed by pouring concrete from the top of the annular grout around the pipe to grade. The concrete well pad should be completed at the same time. Weep holes will be drilled in the base of the guard pipe to facilitate draining of rainwater or purge water from inside the guard pipe.
- Bumper posts or bollards may be necessary for additional well protection, especially in high traffic areas. The bumper posts should be placed around the well pad in a configuration that provides maximum protection to the well and extend a minimum of 3 feet above the ground.

7.4 Double Cased Wells

Under certain site conditions, the use of a double-cased or telescoping (Type III) well may be necessary. Installation of double-cased wells may be required to prevent the interconnection of two separate aquifers, seal off a perched aquifer without creating a vertical hydraulic conduit, prevent cross-contamination during construction of wells in deeper aquifers hydro-stratigraphically below impacted aquifers, or case off highly impacted soils present above the aquifer to prevent potential “dragging down” of contaminants.

Similar to conventional wells, construction of double-cased wells can be accomplished using a varety of drilling methods. Well construction is initiated by “keying” a large diameter, outer casing into a stratigraphic zone of low permeability (clay layer or bedrock). The size of the outer casing should be a minimum of 2 inches greater than the outside diameter of the inner casing to allow installation of annular seal materials during well completion. A pilot borehole should be drilled through the overburden soil and/or contaminated zone into a clay confining layer or bedrock. The borehole for the outer casing should be of sufficient size to contain the outer casing with a minimum of 2 inches around the outside diameter to allow sufficient annular space for tremie or pressure grouting. The boring should extend a minimum of 2 feet into a clay layer and a minimum of 1 foot into bedrock, if possible, to ensure an adequate seal. The boring should never breach a confining layer or keyed zone under any circumstances.

Once the boring is completed, the outer casing can be set in the borehole and sealed with grout. The outer casing can be set two ways, with or without a bottom cap. If no bottom cap is applied, the casing is usually driven approximately 6 inches into the clay confining unit. A grout plug is generally placed in the bottom of the casing and once set, standing water in the casing is evacuated prior to drilling below the casing. As an alternative, a cap can be placed on the bottom of the casing and if set below the water table, the casing can be filled with clean, potable water to hold down the casing in the boring. Grouting should be conducted using tremie-grouting or pressure-grouting methods by pumping grout into the annular space between the outer casing and the borehole wall from the bottom of the casing to the ground surface. Grout around the casing should be allowed to cure at least 24 hours before attempting to drill through the bottom.

Once the grout is cured, a smaller diameter drill pipe/bit is used to bore through the grout plug or bottom cap to the desired well depth. The well is then constructed as described in Section 7.3 above.

7.5 Post Installation Procedures

- Wells should be permanently labelled or marked for identification. Well tags can be used to record the site name, well number, total depth, installation date, etc. At a minimum, the well number will be written in indelible marker or paint on both the outside of the protective casing and inside beneath the casing lid, as well as on the riser pipe.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- A measuring point will be marked on the top of the riser pipe for taking water level measurements. The measuring point can be notched using a knife or saw or can be marked with a waterproof marker or paint. The measuring point will also be the point which will be surveyed for vertical elevation data.
- Upon completion, the following measurements will be taken by the field geologist/engineer and recorded on the well construction diagram.
    - Depth to static water level
    - Depth of non-aqueous phase liquid (NAPL), if present
    - Total depth of well measured from top of casing (TOC)
    - Height of well casing above ground surface
    - Height of protective casing above ground surface
- All monitoring wells will be surveyed for horizontal and vertical control by a licensed surveyor.
- Investigation-derived waste (IDW) including drill cuttings, spent materials (e.g., PPE), and decontamination water should be properly managed in accordance with SOP 3-05, IDW Management.

8.0 Quality Control and Assurance

8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the SAP. Certain quality control (QC) measures should be taken to ensure proper well installation and construction in accordance with this SOP, project specific SAP, and applicable well standards.

8.2 The borehole will be checked for total open depth, and extended by further drilling or shortened by backfilling, as required before installation of the well materials.

8.3 Water level and NAPL presence will be checked during well installation to ensure that the positions of well screen, filter sand, and seals relative to water level conform to project requirements

8.4 The depth to top of each layer of annular materials (i.e., filter sand, bentonite, grout) will be verified and adjusted as necessary for proper placement.

9.0 Records, Data Analysis, Calculations

All field information will be recorded in the field logbook and/or standardized field forms by field personnel. Field data recorded will include drilling contractor information, drilling methods, well material and construction information provided on the boring logs and well construction forms, observations or problems encountered during drilling, fluid level data, and any deviations from the procedures in this SOP and other project plans. Well Construction Forms (Attachment 1) will provide visual and descriptive information the monitoring well and are often the most critical form of documentation generated during the installation of a monitoring well. The field logbook is kept as a general log of activities and should not be used in place of the boring log.

10.0 Attachments or References

10.1 Attachment 1 – Monitoring Well Construction Form

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

10.2 Environmental Protection Agency, United States (EPA). 1987. *A Compendium of Superfund Field Operations Methods*. Office of Solid Waste and Emergency Response. EPA/540/P-87/001.

10.3 EPA. 1990. *Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells*. EPA/600/4-89/034. Office of Research and Development, Washington. March.

10.4 EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

10.5 EPA, 2008. SESD Operating Procedure SESDGUID-101-R0: *Design and Installation of Monitoring Wells*. USEPA, Science and Ecosystem Support Division (SESD), Athens, Georgia. Effective Date February 18, 2008.

10.6 U.S. Army Corps of Engineers. 2008. Manual No. EM 385-1-1. *Safety and Health Requirements*. 15 November 2008. http://140.194.76.129/publications/eng-manuals/em385-1-1/2008_English/toc.html.

10.7 SOP 3-01, *Utility Clearance*.

10.8 SOP 3-05, *IDW Management*

10.9 SOP 3-06, *Equipment Decontamination*.

10.10 SOP 3-16, *Soil and Rock Classification.*

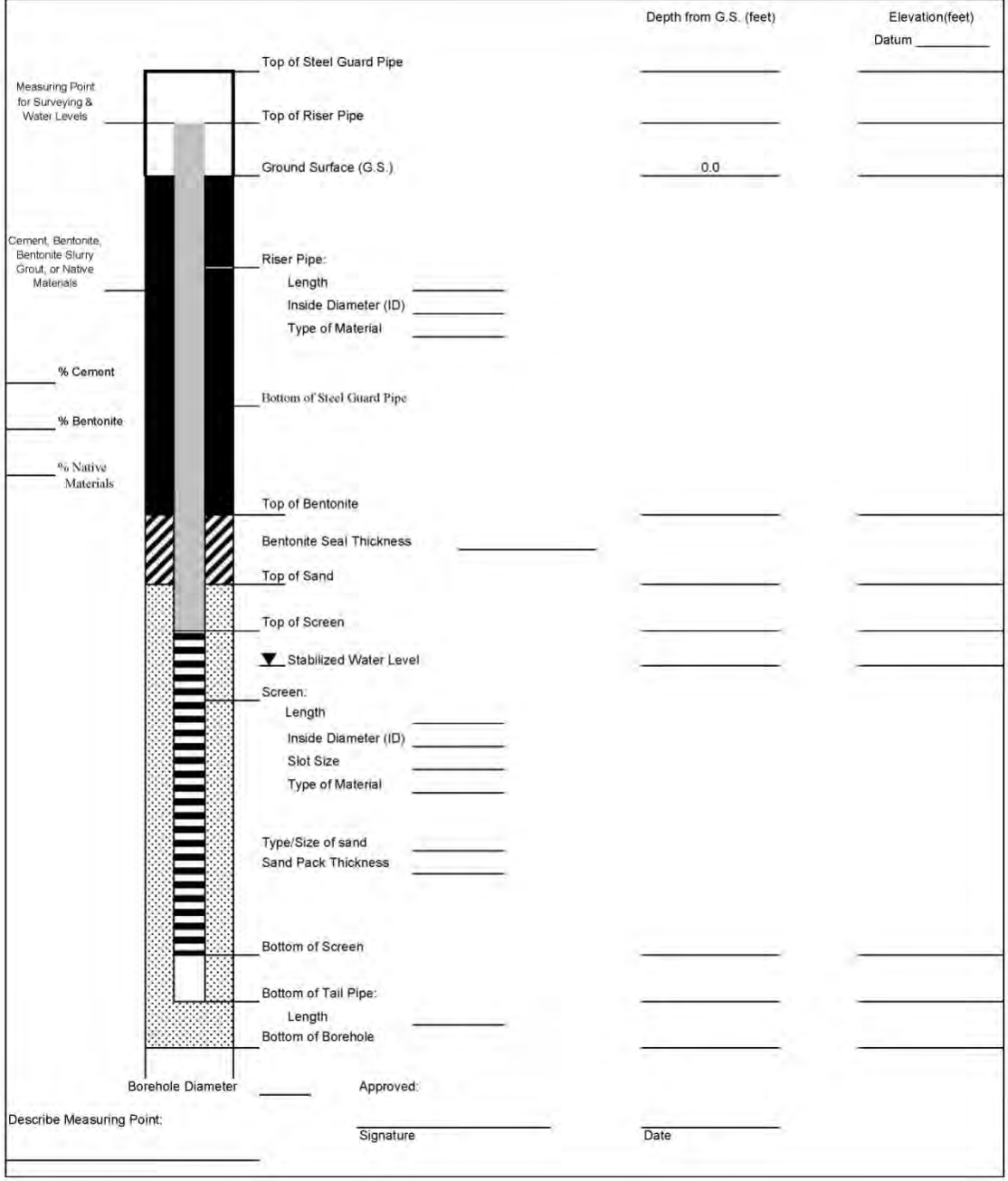
Author	*Reviewer*	*Revisions (Technical or Editorial)*
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 1
Monitoring Well Construction Form

RESOLUTION CONSULTANTS	Client:	**WELL ID:**
	Project Number:	
	Site Location:	Date installed:
	Well Location: Coords:	Inspector:
	Method:	Contractor:

MONITORING WELL CONSTRUCTION DETAIL



PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Monitoring Well Development

Procedure 3-13

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the procedures used for developing newly installed monitoring wells and/or redeveloping existing wells.

1.2 The purpose of well development is to remove interferences from a well to provide better connection between the well and the formation, to improve pumping performance of the well, and to be able to collect more representative information from the well (e.g., samples, test results, etc.). Proper well development will:

- Remove drilling residuals (e.g., water, mud) from the borehole and surrounding formations;
- Improve or restore hydraulic conductivity of the surrounding formations which may have been disturbed during the drilling process;
- Remove residual fines from the well screen and sand pack (filter pack) materials, thus reducing turbidity of groundwater and permitting the collection of more representative groundwater samples.

1.3 There may be circumstances where well development is not desirable, for example, in the presence of non-aqueous phase liquids (NAPL) or other significant contamination if development could worsen the contaminant impact. If NAPL begins to intrude during development, the development process will be halted. This situation will be considered a cause for sample modification requiring approval by the CTO Manager and other stakeholders, as applicable.

1.4 The applicable well development procedures for a particular site may be subject to State or local regulatory requirements. In all cases, the project team should consult their local regulatory requirements and document the selected well development procedure in the project-specific Sampling and Analysis Plan (SAP). For project-specific information refer to the SAP, which takes precedence over these procedures.

1.5 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).

1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) SAP and/or direction from the Site Safety Officer (SSO).

2.2 Monitoring well development may involve chemical hazards associated with potential contaminants in the soil or aquifer being characterized and may involve physical hazards associated with use of well development equipment.

3.0 Terms and Definitions

None.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

4.0 Interferences

4.1 Equipment/materials used for development may react with the groundwater during development. Appropriate development equipment has been selected for the anticipated condition of the groundwater.

4.2 Appropriate development methods such as using a surge-block to flush suspended fines in the groundwater in and out of the well screen can improve the yield of wells and improve their potential to be developed successfully. However, the effectiveness of development can be significantly reduced in wells that do not yield sufficient water to allow this flushing to take place.

4.3 For formations with a significant content of fine-grained materials (silts and clays), or wells with improperly sized screens, it may not be possible to reduce turbidity to commonly acceptable levels. Possible solutions may include collecting a sample even if excessively turbid, or installing a replacement well.

4.4 Development itself disturbs the surrounding formation and disrupts equilibrium conditions within the well. Groundwater samples will not be collected until a minimum of 24 hours after a well is developed to allow conditions to stabilize. For sites with fine-grained formations (silts and clays) and highly sorptive contamination, a longer time period between development and sampling should be considered.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **CTO Manager** is responsible for ensuring that well development activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all personnel involved in well development shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager** is responsible for ensuring that all well development activities are conducted according to the either this procedure or the applicable procedure presented in the project-specific SAP.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure.

5.2.5 The field sampler and/or task manager is responsible for directly supervising the well development procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

6.1 This equipment list was developed to aid in field organization and should be used in planning and preparation. Depending on the site-specific requirements and the development method selected, additional or alternative material and equipment may be necessary. In addition, for sites where groundwater is expected to be contaminated, the materials to be placed down the well and in contact with groundwater should be evaluated so that they are compatible with the chemical conditions expected in the well.

6.2 Equipment and materials used for well development may include, but is not limited to:

Well development equipment

- Surge block

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- Disposable Teflon bailers, appropriate to the diameter of the well(s): 1-inch to 1.5-inch for 2-inch inside diameter (ID) monitoring wells.
- Watterra® footvalve
- Electric submersible pump
- 12-volt power source for electric pump
- High density polyethylene (HDPE) tubing appropriately sized for Watterra® footvalve and/or electric submersible pump
- Drums or containers for storage of purge water
- Nephelometer to measure turbidity
- Multi-parameter water quality meter(s) to measure temperature, pH, conductivity, dissolved oxygen (DO), oxidation reduction potential (ORP)
- Instrument calibration solutions
- Water level meter
- Oil/water interface probe

General equipment

- Project-specific plans including the site-specific HASP and SAP
- Field notebook/field forms/site maps
- Indelible markers/pens
- 5-gallon buckets

Equipment decontamination supplies (refer to SOP 3-06, Equipment Decontamination)

- Health and safety supplies, including personal protective equipment (PPE)
- Appropriate hand tools
- Keys or combinations to access monitoring wells
- Distilled/deionized water supply
- Disposable bailer string (polypropylene)
- Plastic trash bags

7.0 Procedure

Development generally consists of removing water and entrained sediment from the well until the water is clear (to the extent feasible) and the turbidity is reduced, which indicates the well is in good hydraulic connection with the surrounding formation. In addition to simply removing water, development can be improved when flushing through the well screen and gravel pack takes place in both directions, that is, both into the well and into the formation. This action breaks down sediment bridges that can occur in the formation or sand pack, which reduce the connection between the well and the formation

7.1 General Preparation

- All down-well equipment should be decontaminated prior to use and between well locations in accordance with SOP 3-06, Equipment Decontamination
- Although equipment is decontaminated between well locations, if wells are known or suspected to be contaminated based on observations during well installation, it is recommended that well development be conducted in order from the least contaminated to the most contaminated well to minimize the chances of cross-contamination.
- Management of investigation-derived waste (IDW), including development purge water and miscellaneous expendable materials generated during the development process, will be conducted in accordance with SOP 3-05, IDW Management.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- Prior to accessing the well, the wellhead should be cleared of debris and/or standing water. Nothing from the ground surface should be allowed to enter the well.
- The depth to water and total well depth should be measured with a water level meter and recorded in the field logbook or on a Well Development Record (Attachment 1). This information will be used to calculate the volume of standing water (i.e., the well volume) within the well, and plan the specific details of the well development. If wells are suspected to contain NAPL, an oil/water interface probe should be used to measure liquid levels and depth to bottom of the well.
- Permanent monitoring wells will be developed no sooner than 24 hours after well installation is completed in order to allow well completion materials to set properly.

7.2 Monitoring Well Development Procedures

Generally, development will begin by gently surging the well with a surge block or bailer as described in Sections 7.2.1 and 7.2.2, respectively. Surging can become more vigorous as development progresses but initially the well must be gently surged to allow material blocking the screen to become suspended without damaging the well. Next, a bailer can be used to remove the sediment settled at the base of the well. A bailer, Watterra® pump, or electric submersible pump will then be used to purge the well, per Sections 7.2.2, 7.2.3, or 7.2.4, respectively. The well will be purged until the removed water becomes less turbid or per the requirements of the project-specific SAP, or State or local requirements. At this point the well will be surged again with a surge block or bailer. The well can be surged more vigorously at this point. After surging, the well will be purged again until the turbidity once again decreases. The surge/purge cycle should be completed at least three times during the development process. After the last surge, the well will be purged until the development completion criteria outlined in 7.3.2 or per the project-specific SAP are met.

7.2.1 Surge Block

The default method of well development is the use of a surge block in conjunction with pumping or bailing to remove sediment-laden water.

- The construction of the surge block must be appropriate for the diameter of the well. The surge block must be mounted on rods or other stiff materials to extend it to the appropriate depths and to allow for the surge block to be moved up and down in the well.
- Insert the surge block into the well and lower it slowly to the screened or open interval below the static water level. Start the surge action by slowly and gently moving the surge block up and down in the well. A slow initial surging, using plunger strokes of approximately 1 meter or 3 feet, will allow material which is blocking the screen to separate and become suspended.
- After 5 to 10 plunger strokes, remove water from the well using a separate bailer (Section 7.2.2) or pumping techniques (Sections 7.2.3 or 7.2.4). The returned water should be heavily laden with suspended fines. The water will be discharged to 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- In some cases, the bailer or Watterra® foot valve can act as a surge block, flushing water in and out of the well screen as groundwater is removed.
- Repeat the process of surging and pumping/bailing. As development continues, slowly increase the depth of surging to the bottom of the well screen. Surging within the riser portion of the well is neither necessary nor effective.

7.2.2 Bailer

- Tie a string or other cable securely to the bailer. Lower it to the screened or open interval of the monitoring well below the static water level.
- The bailer may be raised and lowered repeatedly within the screened interval to attempt to simulate the action of a surge block by pulling fines through the well screen, and pushing water out into the formation to break down bridging.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- With the bailer full of water, remove it from the well and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- The Watterra® system (Section 7.2.3) or electric submersible pump (Section 7.2.4) may be used as a complementary development method to the bailer, especially when removal of additional water at a faster rate is beneficial.
- Continue alternately surging and bailing, monitoring the purge water periodically (Section 7.3.1) until development completion criteria are met (Section 7.3.2).

7.2.3 Watterra® system

- Attach high-density polyethylene (HDPE) tubing to the decontaminated Watterra® pump foot valve
- Lower the foot valve and tubing assembly near the bottom of the well.
- Lift and lower the tubing to allow water to enter the Watterra® foot valve and travel up the tubing and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- The lifting and lowering action of the Watterra® sysem will cause some surging action to aid in breaking up fine material in the surrounding formation.
- A bailer (Section 7.2.2) may be used as a complementary development method to the Watterra® system, especially during the initial stages of development when a high volume of sediment may be required to be removed.
- An electric submersible pump (Section 7.2.4) may also be used as a complementary development method to the Watterra® system, especially when more volume of water is desired to be pumped or the turbidity criteria cannot be met due to the surging action of the Watterra® system.
- Continue alternately surging and pumping, monitoring the purge water periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

7.2.4 Electric Submersible Pump

- Attach HDPE tubing to the decontaminated electric submersible pump.
- Lower the pump and tubing assembly near the bottom of the well, at least a few inches above the well total depth.
- Begin pumping, discharging the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- Continue alternately surging and pumping, monitoring the purge water discharge periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

7.3 Discharge Monitoring

7.3.1 Monitoring the Progress of Development

The progress of the development is evaluated through visual observation of the suspended sediment load and measurement of the turbidity and other parameters in the purged diischarge water. As development progresses, the water should become clearer, measured turbidity should decrease, and specific capacity (pumping rate divided by drawdown) should stabilize. Water quality parameters, including DO, conductivity, ORP, pH, temperature, and turbidity may be measured and recorded periodically to determine the progress of development using the criteria outlined in Section 7.3.2 or per the project-specific SAP. Water quality parameters should be measured on each well volume removed.

7.3.2 Completion of Development

The well will be considered developed when the following criteria are met or per the criteria set forth in the project-specific SAP:

- A minimum of three times the standing water volume in a well (to include the well screen and casing plus saturated annulus, assuming 30 percent porosity) is removed.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- Groundwater parameters for three consecutive standing water volumes are within the following:
    - pH – within ± 0.2 units
    - Specific conductivity – within ± 3%
    - ORP – within ± 10 mV
    - Temperature – within ±1 degree Celsius
    - Turbidity – at or below 10 nephelometric turbidity units (NTU) or within ± 10% if above 10 NTU.
- The sediment thickness remaining within the well is less than 1 percent of the screen length or less than 30 millimeters (0.1 ft) for screens equal to or less than 10 feet long.

Dissolved oxygen (DO) readings may be recorded but DO readings will not be used as development completion criteria because DO may not stabilize.

If the well has slow groundwater recharge and is purged dry, the well will be considered developed when bailed or pumped dry three times in succession and the turbidity has decreased, or per the requirements set forth in the project-specific SAP. Water quality parameters may be recorded if feasible using the flow-through cell.

If any water is added to the well's borehole during development or drilling, three times the volume of water added will also be removed during well development, or per the requirements set forth in the project-specific SAP.

7.4 Development of Wells with Low Yield

Water is the primary mechanism to remove fines and flush water through the gravel pack for effective development. Therefore, development can be a challenge in wells that do not yield sufficient water to recharge when water is removed. However, often these wells are the most in need of development to improve their performance as they are typically installed in low permeability formations with a high content of fines. Development of these wells can improve their yield.

The surging portion of the development can be successfully performed in a well with standing water regardless of its yield. It is the subsequent removal of fine materials that is hindered when insufficient water is recharged to the well. When wells go dry or drawdown significantly during development, development can be performed intermittently, allowing sufficient water to recharge prior conducting the next stage of surging. These intermittent procedures can take place hours or even days apart, depending on project-specific time constraints.

7.5 Wells containing NAPL

Additional care should be taken when planning development of wells that contain NAPL. If the NAPL is flammable, there are health and safety as well as handling issues to consider. If NAPL in excess of a persistent sheen is noted, the recharge rate will be evaluated through hand bailing. In most cases, it is generally preferable to remove NAPL by bailing to the extent practical prior to performing development. Groundwater parameters, excluding turbidity, will not be collected during well development if NAPL or excessive sheen is noticed in the purged water during development to ensure the meter probes are not fouled or destroyed. Well development will be halted.

Development by surging or pumping the well dry can result in the spreading of NAPL vertically in the soil column around the well. These methods can be used, if information exists describing the vertical thickness of the NAPL smear zone around the well, and if the methods do not result in mounding or drawdown that exceeds this thickness. Alternate methods such as bailing may also be used, but any method should not allow the well to be pumped dry or result in significant drawdown that would spread the NAPL vertically.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

7.6 Temporary Well Points

For certain projects, temporary well points (TWPs) may be installed to collect groundwater samples at a site. Since no sand pack, bentonite chips, or bentonite grout are generally used in the construction of the TWPs, development can proceed as soon as sufficient water has entered the well to static conditions. Due to the small diameter of these wells, generally ¾-inch to 1-inch ID, development will be performed using either a small diameter (0.5-inch) bailer and/or a peristaltic pump with dedicated tubing. The TWPs will have minimal water column and may purge dry during development. However, attempts will be made to remove fines from the well prior to sampling. Purging and sampling may occur as soon as approximately 80% of the static water has re-entered the TWP, or per the requirements set forth in the project-specific SAP.

8.0 Quality Control and Assurance

8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP.

8.2 Quality control (QC) requirements are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for equipment decontamination (frequency and materials) and IDW handling.

9.0 Records, Data Analysis, Calculations

9.1 All data and information (e.g., development method used) must be documented on field data sheets (Attachment 1) or within site logbooks with permanent ink. Data recorded may include the following:

- Well Location
- Weather conditions
- Date and Time
- Purge Method
- Reading/measurements obtained

10.0 Attachments or References

Attachment 1 – Well Development Record

SOP 3-05, *IDW Management*.

SOP 3-06, *Equipment Decontamination*.

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Shawn Dolan Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (June 2012)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 1
Well Development Record

Well/Piezometer Development Record

Well ID:

Client: _____

Project No: _____ Date: _____ Developer: _____

Site Location: _____

Well/Piezometer Data

Well ☐ Piezometer ☐ Diameter _____ Material _____

Measuring Point Description _____ Geology at Screen Interval (if known) _____

Depth to Top of Screen (ft.) _____

Depth to Bottom of Screen (ft.) _____ Time of Water Level Measurement _____

Total Well Depth (ft.) _____ Calculate Purge Volume (gal.) _____

Depth to Static Water Level (ft.) _____ Disposal Method _____

Headspace _____

Original Well Development ☐ Redevelopment ☐ Date of Original Development _____

DEVELOPMENT METHOD _____

PURGE METHOD _____

Time	Total Volume Purged (gal.)	Flow Rate (gpm)	Turbidity (NTU)	Color	pH	Temp	Other

ACCEPTANCE CRITERIA (from workplan)

Minimum Purge Volume Required _____ gallons
Maximum Turbidity Allowed _____ NTUs
Stabilization of parameters _____%

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Has parameters stabilized	☐	☐	☐

If no or N/A explain below:

_____

_____

_____

Signature _____ Date: _____

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Monitoring Well Sampling

Procedure 3-14

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the actions to be used during monitoring well sampling activities and establishes the method for sampling groundwater monitoring wells for water-borne contaminants and general groundwater chemistry. The objective is to obtain groundwater samples that are representative of aquifer conditions with as little alteration to water chemistry as possible.

1.2 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first well. All field sampling personnel responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the well sampling tasks. Conduct preliminary area monitoring of sampling wells to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor phase and liquid matrix through the use of of appropriate personal protective equipment (PPE).

2.2 Observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves and rubberized steel-toed boots. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations. Refer to the project-specific HASP for the required PPE.

2.3 Physical Hazards associated with Well Sampling

- To avoid lifting injuries associated with pump and bailers retrieval, use the large muscles of the legs, not the back.
- Stay clear of all moving equipment, and avoid wearing loose fitting clothing.
- When using tools for cutting purposes, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
- To avoid slip/trip/fall conditions as a result of pump discharge, use textured boots/boot cover bottoms.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
- Be aware of restricted mobility due to PPE.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Potential interferences could result from cross-contamination between samples or sample locations. Minimization of the cross-contamination will occur through the following:

- The use of clean sampling tools at each location as necessary.
- Avoidance of material that is not representative of the media to be sampled.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The **Contract Task Order (CTO) Manager** is responsible for ensuring that monitoring well sampling activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all field sampling personnel involved in monitoring well sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager** is responsible for ensuring that all field sampling personnel follow these procedures.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure.

5.2.5 The field sampler and/or task manager is responsible for directly supervising the groundwater sampling procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

6.1 Purging and Sampling Equipment

- Pump (Peristaltic, Portable Bladder, Submersible)
- Polyethylene or Teflon bladders (for portable bladder pumps)
- Bladder pump controller (for portable bladder pumps)
- Air compressor (for portable bladder pumps)
- Nitrogen cylinders (for portable bladder pumps)
- 12-volt power source
- Polyethylene inlet and discharge tubing (except for VOC analysis which requires Teflon tubing)
- Silicone tubing appropriate for peristaltic pump head
- Teflon bailer appropriately sized for well

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- Disposable bailer string (polypropylene)
- Individual or multi-parameter water quality meter(s) with flow-through cell to measure temperature, pH, specific conductance, dissolved oxygen (DO), oxidation reduction potential (ORP), and/or turbidity
- Turbidity meter
- Water level meter
- Oil/water interface probe

6.2 General Equipment

- Sample kit (i.e., bottles, labels, preservatives, custody records and tape, cooler, ice)
- Sample Chain-of-Custody (COC) forms
- Sample Collection Records
- Sample packaging and shipping supplies
- Waterproof marker or paint
- Distilled/deionized water supply
- Water dispenser bottles
- Flow measurement cup or bucket
- 5-gallon buckets
- Instrument calibration solutions
- Stopwatch or watch
- Disposable Nitrile gloves
- Paper towels
- Trash bags
- Zipper-lock bags
- Equipment decontamination supplies
- Health and safety supplies (as required by the HASP)
- Approved plans such as: project-specific HASP and Sampling and Analysis Plan (SAP)
- Well keys or combinations
- Monitoring well location map(s)
- Field project logbook/pen

7.0 Calibration or Standardization

7.1 Field instruments will be calibrated daily according to the requirements of the SAP and manufacturer's specifications for each piece of equipment. Equipment will be checked daily with the calibration solutions at the end of use of the equipment. Calibration records shall be recorded in the field logbook or appropriate field form.

7.2 If readings are suspected to be inaccurate, the equipment shall be checked with the calibration solutions and/or re-calibrated.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

8.0 Procedure

8.1 Preparation

8.1.1 Site Background Information

Establish a thorough understanding of the purposes of the sampling event prior to field activities. Conduct a review of all available data obtained from the site and pertinent to the water sampling. Review well history data including, but not limited to, well locations, sampling history, purging rates, turbidity problems, previously used purging methods, well installation methods, well completion records, well development methods, previous analytical results, presence of an immiscible phase, historical water levels, and general hydrogeologic conditions.

Previous groundwater development and sampling logs give a good indication of well purging rates and the types of problems that might be encountered during sampling, such as excessive turbidity and low well yield. They may also indicate where dedicated pumps are placed in the water column. To help minimize the potential for cross-contamination, well purging and sampling and water level measurement collection shall proceed from the least contaminated to the most contaminated well as indicated by previous analytical results. This order may be changed in the field if conditions warrant it, particularly if dedicated sampling equipment is used. A review of prior sampling procedures and results may also identify which purging and sampling techniques are appropriate for the parameters to be tested under a given set of field conditions.

8.1.2 Groundwater Analysis Selection

Establish the requisite field and laboratory analyses prior to water sampling. Decide on the types and numbers of quality assurance/quality control (QA/QC) samples to be collected (refer to the project-specific SAP), as well as the type and volume of sample preservatives, the type and number of sample containers, the number of coolers required, and the quantity of ice or other chilling materials. The field sampling personnel shall ensure that the appropriate number and size sample containers are brought to the site, including extras in case of breakage or unexpected field conditions. Refer to the project-specific SAP for the project analytical requirements.

8.2 Groundwater Sampling Procedures

Groundwater sampling procedures at a site shall include:

1) An evaluation of the well security and condition prior to sampling;
2) Decontamination of equipment;
3) Measurement of well depth to groundwater;
4) Assessment of the presence or absence of an immiscible phase;
5) Assessment of purge parameter stabilization;
6) Purging of static water within the well and well bore; and
7) Obtaining a groundwater sample.

Each step is discussed in sequence below. Depending upon specific field conditions, additional steps may be necessary. As a rule, at least 24 hours should separate well development and well sampling events. In all cases, consult the State and local regulations for the site, which may require more stringent time separation between well development and sampling.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

8.2.1 Well Security and Condition

At each monitoring well location, observe the conditions of the well and surrounding area. The following information may be noted on a Groundwater Sample Collection Record (Attachment 1) or in the field logbook:

- Condition of the well's identification marker.
- Condition of the well lock and associated locking cap.
- Integrity of the well – well pad condition, protective outer casing, obstructions or kinks in the well casing, presence of water in the annular space, and the top of the interior casing.
- Condition of the general area surrounding the well.

8.2.2 Decontamination of Equipment

Where possible, dedicated supplies should be used at each well location to minimize the potential for cross-contamination and minimize the amount of investigation derived waste (IDW) fluids resulting from the decontamination process. If decontamination is necessary, establish a decontamination station before beginning sampling. The station shall consist of an area of at least 4 feet by 2 feet covered with plastic sheeting and be located upwind of the well being sampled. The station shall be large enough to fit the appropriate number of wash and rinse buckets, and have sufficient room to place equipment after decontamination. One central cleaning area may be used throughout the entire sampling event. The area around the well being sampled shall also be covered with plastic sheeting to prevent spillage. Further details are presented in SOP 3-06, Equipment Decontamination.

Decontaminate each piece of equipment prior to entering the well. Also, conduct decontamination prior to sampling at a site, even if the equipment has been decontaminated subsequent to its last usage. Additionally, decontaminate each piece of equipment used at the site prior to leaving the site. It is only necessary to decontaminate dedicated sampling equipment prior to installation within the well. Do not place clean sampling equipment directly on the ground or other contaminated surfaces prior to insertion into the well. Dedicated sampling equipment that has been certified by the manufacturer as being decontaminated can be placed in the well without on-site decontamination.

8.2.3 Measurement of Static Water Level Elevation

Before purging the well, measure water levels in all of the wells within the zone of influence of the well being purged. The best practice, if possible, is to measure all site wells (or wells within the monitoring well network) prior to sampling. If the well cap is not vented, remove the cap several minutes before measurement to allow water levels to equilibrate to atmospheric pressure.

Measure the depth to standing water and the total depth of the well to the nearest 0.01 foot to provide baseline hydrologic data, to calculate the volume of water in the well, and to provide information on the integrity of the well (e.g., identification of siltation problems). If not already present, mark an easily identified reference point for water level measurements which will become the measuring point for all water level measurements. This location and elevation must be surveyed.

The device used to measure the water level surface and depth of the well shall be sufficiently sensitive and accurate in order to obtain a measurement to the nearest 0.01 foot reliably. An electronic water level meter will usually be appropriate for this measurement; however, when the groundwater within a particular well is highly contaminated, an inexpensive weighted tape measure can be used to determine well depth to prevent adsorption of contaminants onto the meter tape. The presence of light, non-aqueous phase liquids (LNAPLs) and/or dense, non-aqueous phase liquids (DNAPLs) in a well requires measurement of the elevation of the top and the bottom of the product, generally using an interface probe. Water levels in such wells must then be corrected for density effects to accurately determine the elevation of the water table.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

At each location, measure water levels several times in quick succession to ensure that the well has equilibrated to atmospheric conditions prior to recording the measurement. As stated above, measure all site wells (or wells within the monitoring well network) prior to sampling whenever possible. This will provide a water level database that describes water levels across the site at one time (a synoptic sampling). Prior to sampling, measure the water level in each well immediately prior to purging the well to ascertain that static conditions have been achieved prior to sampling.

8.2.4 Detection of Immiscible Phase Layers

Complete the following steps for detecting the presence of LNAPL and DNAPL before the well is purged for conventional sampling. These procedures may not be required for all wells. Consult the project-specific SAP to determine if assessing the presence of LNAPL and/or DNAPL is necessary.

1) Sample the headspace in the wellhead immediately after the well is opened for organic vapors using either a PID or an organic vapor analyzer, and record the measurements.

2) Lower an interface probe into the well to determine the existence of any immiscible layer(s), LNAPL and/or DNAPL, and record the measurements.

3) Confirm the presence or absence of an immiscible phase by slowly lowering a clear bailer to the appropriate depth, then visually observing the results after sample recovery.

4) In rare instances, such as when very viscous product is present, it may be necessary to utilize hydrocarbon- and water-sensitive pastes for measurement of LNAPL thickness. This is accomplished by smearing adjacent, thin layers of both hydrocarbon- and water-sensitive pastes along a steel measuring tape and inserting the tape into the well. An engineering tape showing tenths and hundredths of feet is required. Record depth to water, as shown by the mark on the water-sensitive paste, and depth to product, as shown by the mark on the product-sensitive paste. In wells where the approximate depth to water and product thickness are not known, it is best to apply both pastes to the tape over a fairly long interval (5 feet or more). Under these conditions, measurements are obtained by trial and error and may require several insertions and retrievals of the tape before the paste-covered interval of the tape encounters product and water. In wells where approximate depths of air-product and product-water interfaces are known, pastes may be applied over shorter intervals. Water depth measurements should not be used in preparation of water table contour maps until they are corrected for depression by the product.

5) If the well contains an immiscible phase, it may be desirable to sample this phase separately. Section 8.2.6 presents immiscible phase sampling procedures. It may not be meaningful to conduct water sample analysis of water obtained from a well containing LNAPLs or DNAPLs. Consult the **CTO Manager** and **Program Quality Manager** if this situation is encountered.

8.2.5 Purging Equipment and Use

General Requirements

The water present in a well prior to sampling may not be representative of in situ groundwater quality and shall be removed prior to sampling. Handle all groundwater removed from potentially contaminated wells in accordance with the IDW handling procedures in SOP 3-05, IDW Management. Purging shall be accomplished by methods as indicated in the project-specific SAP or by those required by State requirements. For the purposes of this SOP, purging methods will be described by removing groundwater from the well using low-flow techniques.

According to the U.S. Environmental Protection Agency (EPA) (EPA, 1996), the rate at which groundwater is removed from the well during purging ideally should be less than 0.2 to 0.3 liters/minute. EPA further states that wells should be purged at rates below those used to develop the well to prevent further development of the well, to prevent damage to the well, and to avoid disturbing accumulated

corrosion or reaction products in the well. EPA also indicates that wells should be purged at or below their recovery rate so that migration of water in the formation above the well screen does not occur.

Realistically, the purge rate should be low enough that substantial drawdown in the well does not occur during purging. In addition, a low purge rate will reduce the possibility of stripping volatile organic compounds (VOCs) from the water, and will reduce the likelihood of increasing the turbidity of the sample due to mobilizing colloids in the subsurface that are immobile under natural flow conditions.

The field sampler shall ensure that purging does not cause formation water to cascade down the sides of the well screen. Wells should not be purged to dryness if recharge causes the formation water to cascade down the sides of the screen, as this will cause an accelerated loss of volatiles. This problem should be anticipated based on the results of either the well development task or historical sampling events. In general, place the intake of the purge pump in the middle of the saturated screened interval within the well to allow purging and at the same time minimize disturbance/overdevelopment of the screened interval in the well. Water shall be purged from the well at a rate that does not cause recharge water to be excessively agitated unless an extremely slow recharging well is encountered where complete evacuation is unavoidable. During the well purging procedure, collect water level and/or product level measurements to assess the hydraulic effects of purging. Sample the well when it recovers sufficiently to provide enough water for the analytical parameters specified. If the well is purged dry, allow the well to recover sufficiently to provide enough water for the specified analytical parameters, and then sample it.

Evaluate water samples on a regular basis during well purging and analyze them in the field preferably using in-line devices (i.e., flow through cell) for temperature, pH, specific conductivity, dissolved oxygen (DO), and oxidation-reduction (redox) potential. Turbidity should be measured separately (outside of the flow-through cell) with a nephelometer or similar device.

Readings should be taken every 2 to 5 minutes during the purging process. These parameters are measured to demonstrate that the natural character of the formation waters has been restored.

Purging shall be considered complete per the requirements set forth in the project-specific SAP, State requirements, or when three consecutive field parameter measurements of temperature, pH, specific conductivity, DO and ORP stabilize within approximately 10 percent and the turbidity is at or below 10 nephelometric turbidity units (NTU) or within ± 10% if above 10 NTU. This criterion may not be applicable to temperature if a submersible pump is used during purging due to the heating of the water by the pump motor. Enter all information obtained during the purging and sampling process into a groundwater sampling log. Attachment 1 shows an example of a groundwater sampling log and the information typically included in the form. Whatever form is used, all blanks need to be completed on the field log during field sampling.

Groundwater removed during purging shall be stored according to the project-specific SAP or per SOP 3-05, IDW Management.

Purging Equipment and Methods

Submersible Pump

A stainless steel submersible pump may be utilized for purging both shallow and deep wells prior to sampling the groundwater for semivolatile and non-volatile constituents, but are generally not preferred for VOCs unless there are no other options (e.g., well over 200 feet deep). For wells over 200 feet deep, the submersible pump is one of the few technologies available to feasibly accomplish purging under any yield conditions. For shallow wells with low yields, submersible pumps are generally inappropriate due to overpumpage of the wells (<1 gallon per minute), which causes increased aeration of the water within the well.

Steam clean or otherwise decontaminate the pump and discharge tubing prior to placing the pump in the well. The submersible pump shall be equipped with an anti-backflow check valve to limit the amount of

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

water that will flow back down the drop pipe into the well. Place the pump in the middle of the saturated screened interval within the well and maintain it in that position during purging.

Bladder Pump

A stainless steel bladder pump can be utilized for purging and sampling wells up to 200 feet in depth for volatile, semivolatile, and non-volatile constituents. Use of the bladder pump is most effective in low to moderate yield wells and are often the preferred method for low-flow sampling. When sampling for VOCs and/or SVOCs, Teflon bladders should be used. Polyethylene bladders may be used when sampling for inorganics.

Either compressed dry nitrogen or compressed dry air, depending upon availability, can operate the bladder pump. The driving gas utilized must be dry to avoid damage to the bladder pump control box. Decontaminate the bladder pump prior to use.

Centrifugal, Peristaltic, or Diaphragm Pump

A centrifugal, peristaltic, or diaphragm pump may be utilized to purge a well if the water level is within 20 feet of ground surface. New or dedicated tubing is inserted into the midpoint of the saturated screened interval of the well. Water should be purged at a rate that satisfies low-flow requirements (i.e., does not cause drawdown). Centrifugal, peristaltic, or diaphragm pump are generally discouraged for VOCs sampling; however, follow methods allowed per the project-specific SAP or State requirements.

Air Lift Pump

Airlift pumps are not appropriate for purging or sampling.

Bailer

Avoid using a bailer to purge a well because it can result in overdevelopment of the well and create excessive purge rates. If a bailer must be used, the bailer should either be dedicated or disposable. Teflon-coated cable mounted on a reel is recommended for lowering the bailer in and out of the well.

Lower the bailer below the water level of the well with as little disturbance of the water as possible to minimize aeration of the water in the well. One way to gauge the depth of water on the reel is to mark the depth to water on the bailer wire with a stainless steel clip. In this manner, less time is spent trying to identify the water level in the well.

8.2.6 Monitoring Well Sampling Methodologies

Sampling Light, Non-Aqueous Phase Liquids (LNAPL)

Collect LNAPL, if present, prior to any purging activities. The sampling device shall generally consist of a dedicated or disposable bailer equipped with a bottom-discharging device. Lower the bailer slowly until contact is made with the surface of the LNAPL, and to a depth less than that of the immiscible fluid/water interface depth as determined by measurement with the interface probe. Allow the bailer to fill with LNAPL and retrieve it.

When sampling LNAPLs, never drop bailers into a well and always remove them from the well in a manner that causes as little agitation of the sample as possible. For example, the bailer should not be removed in a jerky fashion or be allowed to continually bang against the well casing as it is raised. Teflon bailers should always be used when sampling LNAPL. The cable used to raise and lower the bailer shall be composed of an inert material (e.g., stainless steel) or coated with an inert material (e.g., Teflon).

Sampling Dense, Non-Aqueous Phase Liquids (DNAPL)

Collect DNAPL prior to any purging activities. The best method for collecting DNAPL is to use a double-check valve, stainless steel bailer, or a Kemmerer (discrete interval) sampler. The sample shall be collected by slow, controlled lowering of the bailer to the bottom of the well, activation of the closing device, and retrieval.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Groundwater Sampling Methodology

The well shall be sampled when groundwater within it is representative of aquifer conditions per the methods described in Section 8.2.5. Prior to sampling the flow-through cell shall be removed and the samples collected directly from the purge tubing. Flow rates shall not be adjusted once aquifer conditions are met. Additionally, a period of no more than 2 hours shall elapse between purging and sampling to prevent groundwater interaction with the casing and atmosphere. This may not be possible with a slowly recharging well. Measure and record the water level prior to sampling in order to monitor drawdown when using low-flow techniques and gauge well volumes removed and recharged when using non-low-flow techniques.

Sampling equipment (e.g., especially bailers) shall never be dropped into the well, as this could cause aeration of the water upon impact. Additionally, the sampling methodology utilized shall allow for the collection of a groundwater sample in as undisturbed a condition as possible, minimizing the potential for volatilization or aeration. This includes minimizing agitation and aeration during transfer to sample containers, minimizing exposure to sunlight, and immediately placing the sample on ice once collected.

Sampling equipment shall be constructed of inert material. Equipment with neoprene fittings, polyvinyl chloride (PVC) bailers, Tygon® tubing, silicon rubber bladders, neoprene impellers, polyethylene, and Viton® are not acceptable when sampling for organics. If bailers are used, an inert cable/chain (e.g., fluorocarbon resin-coated wire or stainless steel wire or cable) shall be used to raise and lower the bailer. Dedicated equipment is highly recommended for all sampling programs.

Submersible Pumps

The submersible pump must be specifically designed for groundwater sampling (i.e., pump composed of stainless steel and Teflon, sample discharge lines composed of Teflon) and must have a controller mechanism allowing the required low-flow rate. Adjust the pump rate so that flow is continuous and does not pulsate to avoid aeration and agitation within the sample discharge lines. Run the pump for several minutes at the low-flow rate used for sampling to ensure that the groundwater in the lines was obtained at the low-flow rate.

Bladder Pumps

A gas-operated stainless steel bladder pump with adjustable flow control and equipped with a Teflon bladder and Teflon-lined tubing can be effectively utilized to collect a groundwater sample and is considered to be the best overall device for sampling inorganic and organic constituents. If only inorganics are being sampled, polyvinyl bladders and tubing may be used. Operate positive gas displacement bladder pumps in a continuous manner so that they minimize discharge pulsation that can aerate samples in the return tube or upon discharge.

When using a compressor, take several precautions. If the compressor is being powered by a gasoline generator, position the generator downwind of the well. Ground fault circuit interrupters (GFCIs) should always be used when using electric powered equipment. Do not connect the compression hose from the compressor to the pump controller until after the engine has been started.

When all precautions are completed and the compressor has been started, connect the compression hose to the pump controller. Slowly adjust the control knobs to discharge water in the shortest amount of time while maintaining a near constant flow. This does not mean that the compressor must be set to discharge the water as hard as possible. The optimal setting is one that produces the largest volume of purge water per minute (not per purge cycle) while maintaining a near constant flow rate.

Prior to sampling, adjust the flow rate (purge rate) to yield 100 to 300 mL/minute. Avoid settings that produce pulsating streams of water instead of a steady stream if possible. Operate the pump at this low flow rate for several minutes to ensure that drawdown is not occurring. At no time shall the sample flow rate exceed the flow rate used while purging.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

For those samples requiring filtration, it is recommended to use an in-line high capacity filter after all non-filtered samples have been collected.

***Peristaltic Pumps*:**

A peristaltic pump is a type of positive displacement pump that moves water via the process of peristalsis. The pump uses a flexible hose fitted inside a circular pump casing. A rotor with cams compresses the flexible tube as the rotor turns, which forces the water to be pumped to move through the tube. In peristaltic pumps, no moving parts of the pump are in contact with the water being pumped. Displacement is determined by tube size, so delivery rate can only be changed during operation by varying pump speed. Peristaltic pumps are simple and quite inexpensive for the flow rates they provide.

There are several methods available for transferring the sample into the laboratory containers. The selected method may vary based on State requirements and should be documented in the project-specific SAP. Samples typically can be collected directly from the discharge end of the Teflon tubing, after it has been disconnected from the flow through cell. For volatile analyses, the sampler should make sure that the pump is set such that a smooth laminar flow is achieved. In all cases, the project team should consult their local regulatory requirements and document the selected sample collection procedure in the project-specific SAP.

Bailers

A single- or double-check valve Teflon or stainless steel bailer equipped with a bottom discharging device can be utilized to collect groundwater samples. Bailers have a number of disadvantages, however, including a tendency to alter the chemistry of groundwater samples due to degassing, volatilization, and aeration; the possibility of creating high groundwater entrance velocities; differences in operator techniques resulting in variable samples; and difficulty in determining where in the water column the sample was collected. Therefore, use bailers for groundwater sampling only when other types of sampling devices cannot be utilized for technical, regulatory, or logistical reasons.

Dedicated or disposable bailers should always be used in order to eliminate the need for decontamination and to limit the potential of cross-contamination. Each time the bailer is lowered to the water table, lower it in such a way as to minimize disturbance and aeration of the water column within the well.

8.2.7 Sample Handling and Preservation

Many of the chemical constituents and physiochemical parameters to be measured or evaluated during groundwater monitoring programs are chemically unstable and require preservation. The U.S. EPA document entitled, *Test Methods for Evaluating Solid Waste – Physical/Chemical Methods (SW-846)* (EPA 1997), includes a discussion of appropriate sample preservation procedures. In addition, SW-846 provides guidance on the types of sample containers to use for each constituent or common set of parameters. In general, check with specific laboratory or State requirements prior to obtaining field samples. In many cases, the laboratory will supply the necessary sample bottles and required preservatives. In some cases, the field sampling personnel may add preservatives in the field.

Improper sample handling may alter the analytical results of the sample. Therefore, transfer samples in the field from the sampling equipment directly into the container that has been prepared specifically for that analysis or set of compatible parameters as described in the project-specific SAP. It is not an acceptable practice for samples to be composited in a common container in the field and then split in the laboratory, or poured first into a wide mouth container and then transferred into smaller containers.

Collect groundwater samples and place them in their proper containers in the order of decreasing volatility and increasing stability. A preferred collection order for some common groundwater parameters is:

1. VOCs and total organic halogens (TOX)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

2. Dissolved gases, total organic carbon (TOC), total fuel hydrocarbons
3. Semivolatile organics, pesticides
4. Total metals, general minerals (unfiltered)
5. Dissolved metals, general minerals (filtered)
6. Phenols
7. Cyanide
8. Sulfate and chloride
9. Nitrate and ammonia
10. Radionuclides

When sampling for VOCs, collect water samples in vials or containers specifically designed to prevent loss of VOCs from the sample. The analytical laboratory performing the analysis shall provide these vials. Collect groundwater from the sampling device in vials by allowing the groundwater to slowly flow along the sides of the vial. Sampling equipment shall not touch the interior of the vial. Fill the vial above the top of the vial to form a positive meniscus with no overflow. No headspace shall be present in the sample container once the container has been capped. This can be checked by inverting the bottle once the sample is collected and tapping the side of the vial to dislodge air bubbles. Sometimes it is not possible to collect a sample without air bubbles, particularly water that has high concentrations of dissolved gasses. In these cases, the field sampling personnel shall document the occurrence in the field logbook and/or sampling worksheet at the time the sample was collected. Likewise, the analytical laboratory shall note in the laboratory analysis reports any headspace in the sample container(s) at the time of receipt by the laboratory.

Special Handling Considerations

In general, samples for organic analyses should not be filtered. However, high turbidity samples for PCB analysis may require filtering. Consult the project-specific SAP for details on filtering requirements. Samples shall not be transferred from one container to another because this could cause aeration or a loss of organic material onto the walls of the container. TOX and TOC samples should be handled in the same manner as VOC samples.

When collecting total and dissolved metals samples, the samples should be collected sequentially. The total metals sample is collected from the pump unfiltered. The dissolved metals sample is collected after filtering with a 0.45-micron membrane in-line filter. Allow at least 500 mL of effluent to flow through the filter prior to sampling to ensure that the filter is thoroughly wetted and seated in the filter capsule. If required by the project-specific SAP, include a filter blank for each lot of filters used and always record the lot number of the filters.

Field Sampling Preservation

Preserve samples immediately upon collection. Ideally, sampling containers will be pre-preserved with a known concentration and volume of preservative. Certain matrices that have alkaline pH (greater than 7) may require more preservative than is typically required. An early assessment of preservation techniques, such as the use of pH strips after initial preservation, may therefore be appropriate. Guidance for the preservation of environmental samples can be found in the U.S. EPA *Handbook for Sampling and Sample Preservation of Water and Wastewater* (EPA 1982). Additional guidance can be found in other U.S. EPA documents (EPA 1992, 1996).

Field Sampling Log

A groundwater sampling log provided as Attachment 1 shall document the following:

- Identification of well

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- Well depth
- Static water level depth and measurement technique
- Presence of immiscible layers and detection method
- Well yield
- Purge volume and pumping rate
- Time that the well was purged
- Sample identification numbers
- Well evacuation procedure/equipment
- Sample withdrawal procedure/equipment
- Date and time of collection
- Types of sample containers used
- Preservative(s) used
- Parameters requested for analysis
- Field analysis data
- Field observations on sampling event
- Name of sampler
- Weather conditions

9.0 Quality Control and Assurance

9.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.

9.2 Quality control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for sample preservation and holding times, container types, sample packaging and shipment, as well as requirements for the collection of various QC samples such as trip blanks, field blanks, equipment rinse blanks, and field duplicate samples.

10.0 Data and records management

10.1 Records will be maintained in accordance with SOP 3-03, Recordkeeping, Sample Labelling, and Chain-of-Custody. Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:

- Sample Collection Records;
- Field logbook;
- Chain-of-custody forms; and
- Shipping labels.

10.2 Sample collection records (Attachment 1) will provide descriptive information for the purging process and the samples collected at each monitoring well.

10.3 The field logbook is kept as a general log of activities and should not be used in place of the sample collection record.

10.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.

10.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

11.0 Attachments or References

Attachment 1 – Groundwater Sampling Collection Record

ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites.* ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. www.astm.org.

Environmental Protection Agency, United States (EPA). 1982. *Handbook for Sampling and Sample Preservation of Water and Wastewater.* EPA-600/4-82-029. Cincinnati: EPA Office of Research and Development, Environmental Monitoring and Support Laboratory.

EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

EPA. 1996. *Ground Water Issue: Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*. EPA/540/S-95/504. Office of Solid Waste and Emergency Response. April.

EPA. 1997. *Test Methods for Evaluating Solid Waste, Physical/Chemical Method (SW-846)*. 3rd ed., Final Update IIIA. Office of Solid Waste. Online updates at: http://www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

NAVSEA T0300-AZ-PRO-010. *Navy Environmental Compliance Sampling and Field Testing Procedures Manual*. August 2009.

SOP 3-03, *Recordkeeping, Sample Labelling, and Chain-of-Custody*.

SOP 3-05, *IDW Management.*

SOP 3-06, *Equipment Decontamination*.

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 1
Groundwater Sample Collection Record

Well ID:

Groundwater Sample Collection Record

Client: _____ Date: _____ Time: Start _____ am/pm
Project No: _____ Finish _____ am/pm
Site Location: _____
Weather Conds: _____ Collector(s): _____

1. WATER LEVEL DATA: (measured from Top of Casing)

a. Total Well Length _____ c. Length of Water Column _____ (a-b)

Casing Diameter/Material _____

b. Water Table Depth _____ d. Calculated Well Volume (see back) _____

2. WELL PURGEABLE DATA

a. Purge Method: _____

b. Acceptance Criteria defined (see SAP or Work Plan)
- Minimum Required Purge Volume (@ _____ well volumes) _____
- Maximum Allowable Turbidity _____ NTUs
- Stabilization of parameters _____ %

c. Field Testing Equipment used: Make Model Serial Number

_____
_____
_____

Time (min)	Volume Removed (gal)	Temp. (°C)	pH s.u.	Spec. Cond. (µS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (m)	Color/Odor/etc.

d. Acceptance criteria pass/fail Yes No N/A (continued on back)

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Have parameters stabilized	☐	☐	☐

If no or N/A - Explain below.

_____

3. SAMPLE COLLECTION: Method: _____

Sample ID	Container Type	No. of Containers	Preservation	Analysis Req.	Time

Comments _____

Signature _____ Date _____

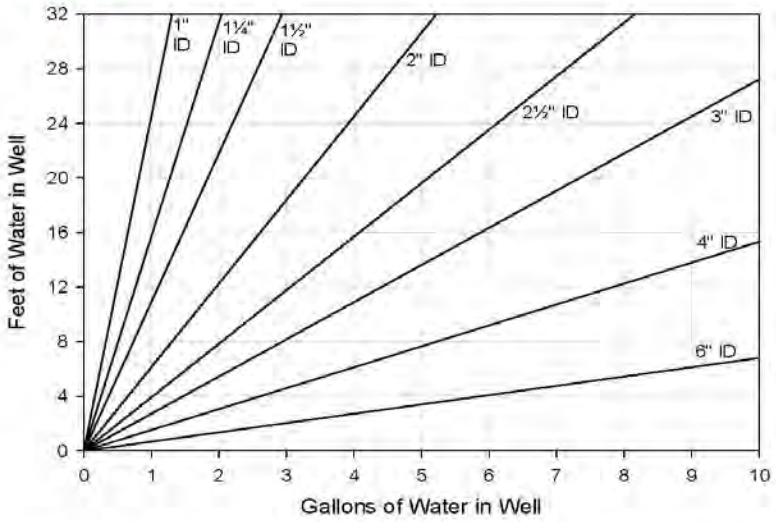
Page 1 of 2

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Purge Volume Computation

RESOLUTION CONSULTANTS

Well ID:



Volume / Linear Ft. of Pipe		
ID (in)	Gallon	Liter
¼	0.0025	0.0097
⅜	0.0057	0.0217
½	0.0102	0.0386
¾	0.0229	0.0869
1	0.0408	0.1544
1¼	0.0637	0.2413
1½	0.0918	0.3475
2	0.1632	0.6178
2½	0.2550	0.9653
3	0.3672	1.3900
4	0.6528	2.4711
6	1.4688	5.5600

(continued from front)

Time (min)	Volume Removed (gal)	Temp. (°C)	pH s.u.	Spec. Cond. (µS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (m)	Color/Odor/etc.

Signature _____ Date _____

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Soil and Rock Classification

Procedure 3-16

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) to thoroughly describe the physical characteristics of the sample and classify it according to the Unified Soil Classification System (USCS).

1.2 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific SAP.

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling. All **field sampling personnel** responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety Officer (SSO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the HASP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSO.

2.4 The health and safety considerations for the work associated with soil classification include:

- At no time during classification activities are personnel to reach for debris near machinery that is in operation, place any samples in their mouth, or come in contact with the soils/rocks without the use of gloves.
- Stay clear of all moving equipment and be aware of pinch points on machinery. Avoid wearing loose fitting clothing.
- When using cutting tools, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
- To avoid heat/cold stress as a results of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and in case of extreme cold, wear insulating clothing.

3.0 Terms and Definitions

None.

4.0 Interference

None.

5.0 Training and Qualifications

5.1 The **Contract Task Order (CTO) Manager** is responsible for ensuring that the soil and rock classification procedures comply with this procedure. The **CTO Manager** is responsible for ensuring that all personnel involved in soil and rock classification shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.3 The **Field Manager** is responsible for ensuring that all project **field personnel** follow these procedures.

5.4 Field personnel are responsible for the implementation of this procedure. Minimum qualifications for **field sampling personnel** require that one individual on the field team shall have a minimum of 6 months of experience with soil and rock classification.

5.5 The **project geologist** and/or **task manager** is responsible for directly supervising the soil and rock classification procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the **Program Quality Manager** and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

6.1 The following equipment list contains materials which may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Personal protective equipment (PPE) and other safety equipment, as required by the HASP
- Field log book and pen with indelible ink
- Boring log

- Munsell Soil Color Chart
- Scoopula, spatula, and/or other small hand tools
- California Sampler
- Hand-held penetrometer

7.0 Calibration or Standardization

None.

8.0 Procedure

8.1 Soil Classification

The basic purpose of the classification of soil is to thoroughly describe the physical characteristics of the sample and to classify it according to an appropriate soil classification system. The USCS was developed so that soils could be described on a common basis by different investigators and serve as a "shorthand" description of soil. A classification of a soil in accordance with the USCS includes not only a group symbol and name, but also a complete word description.

Describing soil on a common basis is essential so that soil described by different site qualified personnel is comparable. Site individuals describing soil as part of site activities *must* use the classification system described herein to provide the most useful geologic database for all present and future subsurface investigations and remedial activities.

The site geologist or other qualified individual shall describe the soil and record the description in a boring log, logbook, and/or electronic field data collection device. The essential items in any written soil description are as follows:

- Classification group name (e.g., silty sand)
- Color, moisture, and odor
- Range of particle sizes and maximum particle size
- Approximate percentage of boulders, cobbles, gravel, sand, and fines
- Plasticity characteristics of the fines
- In-place conditions, such as consistency, density, and structure
- USCS classification symbol

The USCS serves as ”shorthand“ for classifying soil into 15 basic groups:

GW[1] Well graded (poorly sorted) gravel (>50 percent gravel, <5percent fines)

GP[1] Poorly graded (well sorted) gravel (>50percent gravel, <5percent fines)

GM[1] Silty gravel (>50 percent gravel, >15 percent silt)

GC[1] Clayey gravel (>50 percent gravel, >15 percent clay)

SW[1] Well graded (poorly sorted) sand (>50 percent sand, <5 percent fines)

SP[1] Poorly graded (well sorted) sand (>50 percent sand, <5 percent fines)

[1] If percentage of fine is 5 percent to 15 percent, a dual identification shall be given (e.g., a soil with more than 50 percent poorly sorted gravel and 10 percent clay is designated GW-GC.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

SM[1] Silty sand (>50 percent sand, >15 percent silt)

SC[1] Clayey sand (>50 percent sand, >15 percent clay)

ML[2] Inorganic, low plasticity silt (slow to rapid dilatancy, low toughness, and plasticity)

CL[2] Inorganic, low plasticity (lean) clay (no or slow dilatancy, medium toughness and plasticity)

MH[2] Inorganic elastic silt (no to slow dilatancy, low to medium toughness and plasticity)

CH[2] Inorganic, high plasticity (fat) clay (no dilatancy, high toughness, and plasticity)

OL Organic low plasticity silt or organic silty clay

OH Organic high plasticity clay or silt

PT Peat and other highly organic soil

Figure8-1 defines the terminology of the USCS. Flow charts presented in Figure 8-2 and indicate the process for describing soil. The particle size distribution and the plasticity of the fines are the two properties of soil used for classification. In some cases, it may be appropriate to use a borderline classification (e.g., SC/CL) if the soil has been identified as having properties that do not distinctly place the soil into one group.

8.1.1 Estimation of Particle Size Distribution

One of the most important factors in classifying a soil is the estimated percentage of soil constituents in each particle size range. Being proficient in estimating this factor requires extensive practice and frequent checking. The steps involved in determining particle size distribution are listed below:

1. Select a representative sample (approximately 1/2 of a 6-inch long by 2.5-inch diameter sample liner).
2. Remove all particles larger than 3 inches from the sample. Estimate and record the percent by volume of these particles. Only the fraction of the sample smaller than 3 inches is classified.
3. Estimate and record the percentage of dry mass of gravel (less than 3 inches and greater than 1/4 inch).
4. Considering the rest of the sample, estimate, and record the percentage of dry mass of sand particles (about the smallest particle visible to the unaided eye).
5. Estimate and record the percentage of dry mass of fines in the sample (do not attempt to separate silts from clays).
6. Estimate percentages to the nearest 5 percent. If one of the components is present in a quantity considered less than 5 percent, indicate its presence by the term “trace”.
7. The percentages of gravel, sand, and fines must add up to 100 percent. “Trace” is not included in the 100 percent total.

8.1.2 Soil Dilatancy, Toughness, and Plasticity

8.1.2.1 Dilatancy

To evaluate dilatancy, follow these procedures:

[2] If the soil is estimated to have 15 percent to 25 percent sand or gravel, or both, the words “with sand” or “with gravel” (whichever predominates) shall be added to the group name (e.g., clay with sand, CL; or silt with gravel, ML). If the soil is estimated to have 30 percent or more sand or gravel, or both, the words “sandy” or “gravely” (whichever predominates) shall be added to the group name (e.g., sandy clay, CL). If the percentage of sand is equal to the percent gravel, use “sandy.”

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

1. From the specimen, select enough material to mold into a ball about 1/2 inch (12 millimeters [mm]) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.
2. Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 8-1. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

Table 8-1: Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in specimen.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

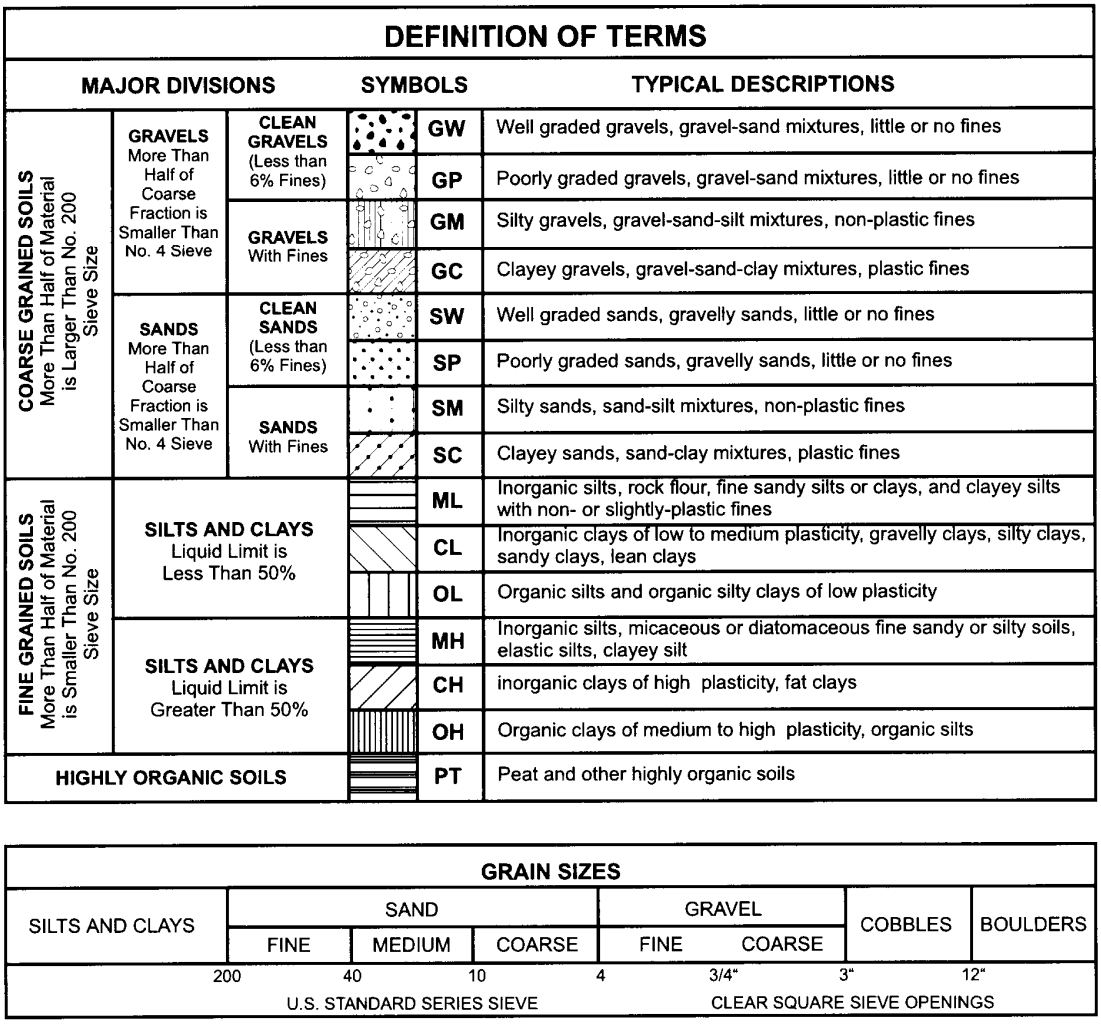
8.1.2.2 Toughness

Following the completion of the dilatancy test, shape the test specimen into an elongated pat and roll it by hand on a smooth surface or between the palms into a thread about 1/8 inch (3 mm) in diameter. (If the sample is too wet to roll easily, spread it into a thin layer and allow it to lose some water by evaporation.) Fold the sample threads and re-roll repeatedly until the thread crumbles at a diameter of about 1/8 inch. The thread will crumble at a diameter of 1/8 inch when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, lump the pieces together and knead it until the lump crumbles. Note the toughness of the material during kneading. Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 8-2.

Table 8-2: Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread near the plastic limit. The thread and the lump have very high stiffness.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.



DEFINITION OF TERMS					
MAJOR DIVISIONS				SYMBOLS	TYPICAL DESCRIPTIONS
COARSE GRAINED SOILS More Than Half of Material is Larger Than No. 200 Sieve Size	GRAVELS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN GRAVELS (Less than 6% Fines)		GW	Well graded gravels, gravel-sand mixtures, little or no fines
				GP	Poorly graded gravels, gravel-sand mixtures, little or no fines
		GRAVELS With Fines		GM	Silty gravels, gravel-sand-silt mixtures, non-plastic fines
				GC	Clayey gravels, gravel-sand-clay mixtures, plastic fines
	SANDS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN SANDS (Less than 6% Fines)		SW	Well graded sands, gravelly sands, little or no fines
				SP	Poorly graded sands, gravelly sands, little or no fines
		SANDS With Fines		SM	Silty sands, sand-silt mixtures, non-plastic fines
				SC	Clayey sands, sand-clay mixtures, plastic fines
FINE GRAINED SOILS More Than Half of Material is Smaller Than No. 200 Sieve Size	SILTS AND CLAYS Liquid Limit is Less Than 50%			ML	Inorganic silts, rock flour, fine sandy silts or clays, and clayey silts with non- or slightly-plastic fines
				CL	Inorganic clays of low to medium plasticity, gravelly clays, silty clays, sandy clays, lean clays
				OL	Organic silts and organic silty clays of low plasticity
	SILTS AND CLAYS Liquid Limit is Greater Than 50%			MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts, clayey silt
				CH	inorganic clays of high plasticity, fat clays
				OH	Organic clays of medium to high plasticity, organic silts
HIGHLY ORGANIC SOILS				PT	Peat and other highly organic soils

GRAIN SIZES								
SILTS AND CLAYS	SAND			GRAVEL		COBBLES	BOULDERS	
	FINE	MEDIUM	COARSE	FINE	COARSE			
200	40	10	4	3/4"	3"	12"		
U.S. STANDARD SERIES SIEVE				CLEAR SQUARE SIEVE OPENINGS				

Figure8-1: Unclassified Soil Classification System (USCS)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

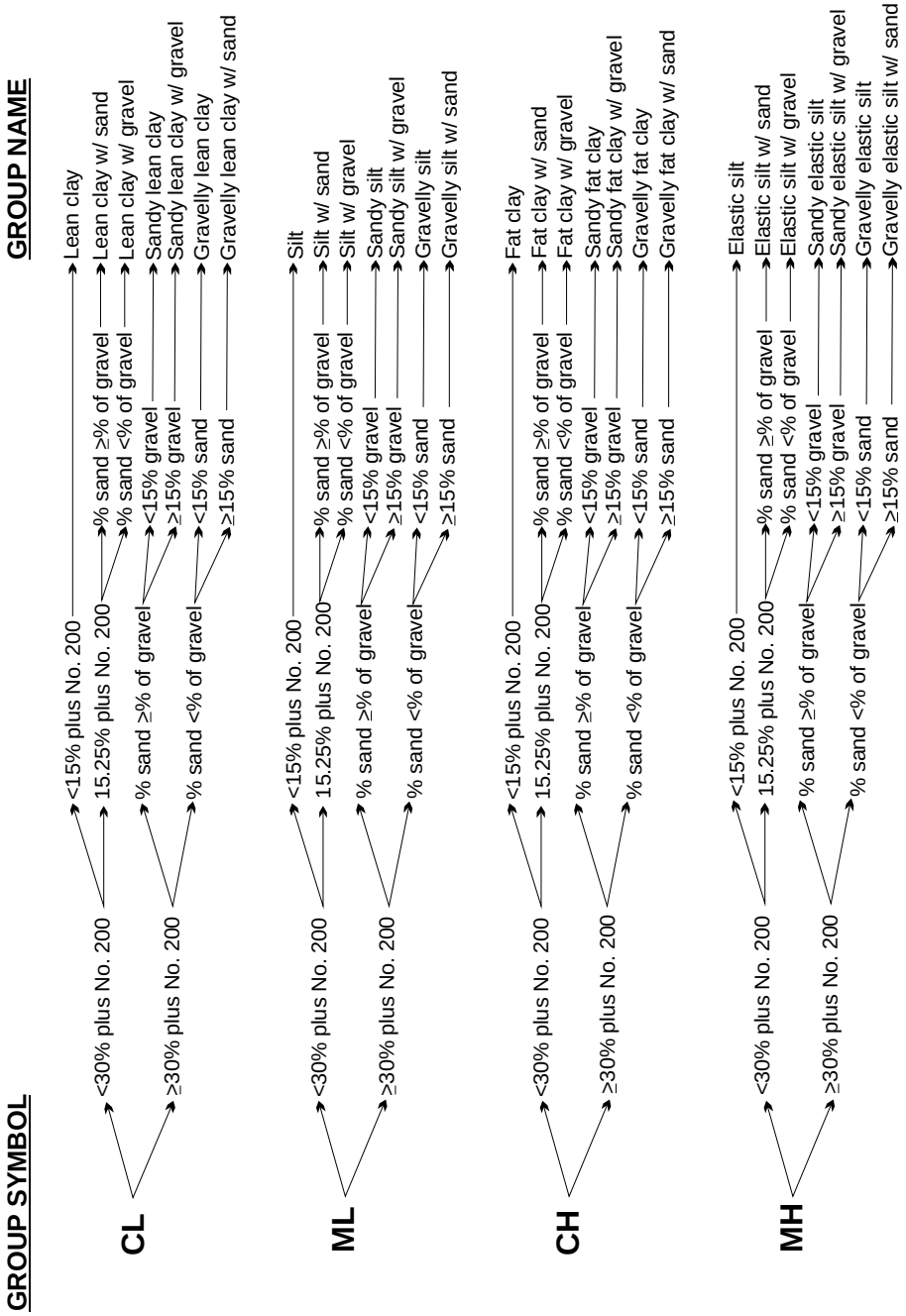


Figure 8-2: Flow Chart for Fine Grain Soil Classification

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

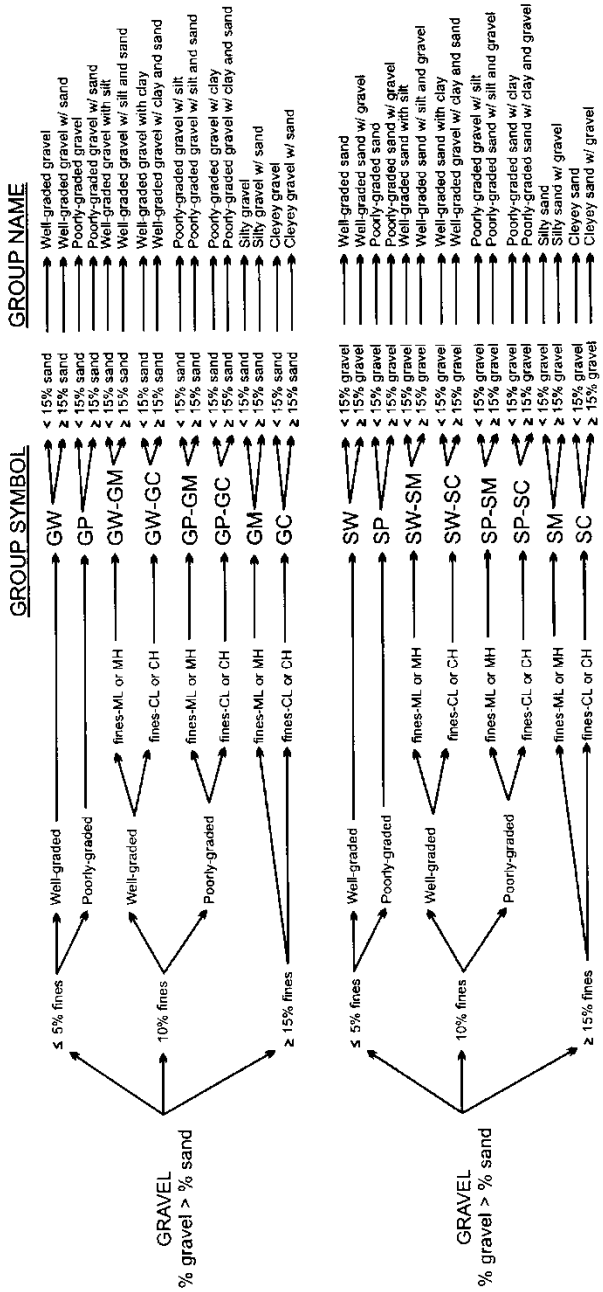


Figure 8-3: Flow Chart for Soil with Gravel

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

8.1.2.3 Plasticity

The plasticity of a soil is defined by the ability of the soil to deform without cracking, the range of moisture content over which the soil remains in a plastic state, and the degree of cohesiveness at the plastic limit. The plasticity characteristic of clays and other cohesive materials is defined by the liquid limit and plastic limit. The liquid limit is defined as the soil moisture content at which soil passes from the liquid to the plastic state as moisture is removed. The test for the liquid limit is a laboratory, not a field, analysis.

The plastic limit is the soil moisture content at which a soil passes from the plastic to the semi-solid state as moisture is removed. The plastic limit test can be performed in the field and is indicated by the ability to roll a 1/8-inch (0.125-inch) diameter thread of fines, the time required to roll the thread, and the number of times the thread can be re-rolled when approaching the plastic limit.

The plasticity tests are not based on natural soil moisture content, but on soil that has been thoroughly mixed with water. If a soil sample is too dry in the field, add water prior to performing classification. If a soil sample is too sticky, spread the sample thin and allow it to lose some soil moisture.

Table 8-3 presents the criteria for describing plasticity in the field using the rolled thread method.

Table 8-3: Criteria for Describing Plasticity

Description	Criteria
Non-Plastic	A 1/8-inch thread cannot be rolled.
Low Plasticity	The thread can barely be rolled.
Medium Plasticity	The thread is easy to roll and not much time is required to reach the plastic limit.
High Plasticity	It takes considerable time rolling the thread to reach the plastic limit.

8.1.3 **Angularity**

The following criteria describe the angularity of the coarse sand and gravel particles:

- **Rounded** particles have smoothly-curved sides and no edges.
- **Subrounded** particles have nearly plane sides, but have well-rounded corners and edges.
- **Subangular** particles are similar to angular, but have somewhat rounded or smooth edges.
- **Angular** particles have sharp edges and relatively plane sides with unpolished surfaces. Freshly broken or crushed rock would be described as angular.

8.1.4 **Color, Moisture, and Odor**

The natural moisture content of soil is very important. Table **8-4** shows the terms for describing the moisture condition and the criteria for each.

Table 8-4: Soil Moisture Content Qualifiers

Qualifier	Criteria
Dry	Absence of moisture, dry to the touch
Moist	Damp but no visible water
Wet	Visible water, usually soil is below water table

Color is described by hue and chroma using the Munsell Soil Color Chart (Munsell 2000). For uniformity, all site geologists shall utilize this chart for soil classification. Doing so will facilitate correlation of geologic units between boreholes logged by different geologists. The Munsell Color Chart is a small booklet of numbered color chips with names like “5YR 5/6, yellowish-red.” Note mottling or banding of colors. It is particularly important to note and describe staining because it may indicate contamination.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

In general, wear a respirator if strong organic odors are present. If odors are noted, describe them if they are unusual or suspected to result from contamination. An organic odor may have the distinctive smell of decaying vegetation. Unusual odors may be related to hydrocarbons, solvents, or other chemicals in the subsurface. An organic vapor analyzer may be used to detect the presence of volatile organic contaminants.

8.1.5 In-Place Conditions

Describe the conditions of undisturbed soil samples in terms of their density/consistency (i.e., compactness), cementation, and structure utilizing the following guidelines:

8.1.5.1 Density/Consistency

Density and consistency describe a physical property that reflects the relative resistance of a soil to penetration. The term “density” is commonly applied to coarse to medium-grained sediments (i.e., gravels, sands), whereas the term “consistency” is normally applied to fine-grained sediments (i.e., silts, clays). There are separate standards of measure for both density and consistency that are used to describe the properties of a soil.

The density or consistency of a soil is determined by observing the number of blows required to drive a 1 3/8-inch (35 mm) diameter split barrel sampler 18 inches using a drive hammer weighing 140 lbs (63.5 kilograms [kg]) dropped over a distance of 30 inches (0.76 meters). Record the number of blows required to penetrate each 6 inches of soil in the field boring log during sampling. The first 6 inches of penetration is considered to be a seating drive; therefore, the blow count associated with this seating drive is recorded, but not used in determining the soil density/consistency. The sum of the number of blows required for the second and third 6 inches of penetration is termed the “standard penetration resistance,” or the “N-value.” The observed number of blow counts must be corrected by an appropriate factor if a different type of sampling device (e.g., Modified California Sampler with liners) is used. For a 2 3/8-inch inner diameter (I.D.) Modified California Sampler equipped with brass or stainless steel liners and penetrating a cohesionless soil (sand/gravel), the N-value from the Modified California Sampler must be divided by 1.43 to provide data that can be compared to the 1 3/8-inch diameter sampler data.

For a cohesive soil (silt/clay), the N-value for the Modified California Sampler should be divided by a factor of 1.13 for comparison with 1 3/8-inch diameter sampler data.

Drive the sampler and record blow counts for each 6-inch increment of penetration until one of the following occurs:

- A total of 50 blows have been applied during any one of the three 6-inch increments; a 50-blow count occurrence shall be termed “refusal” and noted as such on the boring log.
- A total of 150 blows have been applied.
- The sampler is advanced the complete 18 inches without the limiting blow counts occurring, as described above.

If the sampler is driven less than 18 inches, record the number of blows per partial increment on the boring log. If refusal occurs during the first 6 inches of penetration, the number of blows will represent the N-value for this sampling interval. Table 8-5 and Table 8-6 present representative descriptions of soil density/consistency vs. N-values.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Table 8-5: Measuring Soil Density with a California Sampler – Relative Density (Sands, Gravels)

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.43 factor
Very Loose	0–4	0–6
Loose	4–10	6–14
Medium Dense	10–30	14–43
Dense	30–50	43–71
Very Dense	> 50	> 71

Table 8-6: Measuring Soil Density with a California Sampler – Fine Grained Cohesive Soil

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.13 factor
Very Soft	0–2	0–2
Soft	2–4	2–4
Medium Stiff	4–8	4–9
Stiff	8–16	9–18
Very Stiff	16–32	18–36
Hard	> 32	> 36

For undisturbed fine-grained soil samples, it is also possible to measure consistency with a hand-held penetrometer. The measurement is made by placing the tip of the penetrometer against the surface of the soil contained within the sampling liner or shelby tube, pushing the penetrometer into the soil a distance specified by the penetrometer manufacturer, and recording the pressure resistance reading in pounds per square foot (psf). The values are as follows (Table 8-7):

Table 8-7: Measuring Soil Consistency with a Hand-Held Penetrometer

Description	Pocket Penetrometer Reading (psf)
Very Soft	0–250
Soft	250–500
Medium Stiff	500–1000
Stiff	1000–2000
Very Stiff	2000–4000
Hard	>4000

Consistency can also be estimated using thumb pressure using Table 8-8.

Table 8-8: Measuring Soil Consistency Using Thumb Pressure

Description	Criteria
Very Soft	Thumb will penetrate soil more than 1 inch (25 mm)
Soft	Thumb will penetrate soil about 1 inch (25 mm)
Firm	Thumb will penetrate soil about 1/4 inch (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

8.1.5.2 *Cementation*

Cementation is used to describe the friability of a soil. Cements are chemical precipitates that provide important information as to conditions that prevailed at the time of deposition, or conversely, diagenetic effects that occurred following deposition. Seven types of chemical cements are recognized by Folk (1980). They are as follows:

- Quartz – siliceous
- Chert – chert-cemented or chalcedonic
- Opal – opaline
- Carbonate – calcitic, dolomitic, sideritic (if in doubt, calcareous should be used)
- Iron oxides – hematitic, limonitic (if in doubt, ferruginous should be used)
- Clay minerals – if the clay minerals are detrital or have formed by recrystallization of a previous clay matrix, they are not considered to be a cement. Only if they are chemical precipitates, filling previous pore space (usually in the form of accordion-like stacks or fringing radial crusts) should they be included as “kaolin-cemented,” “chlorite-cemented,” etc.
- Miscellaneous minerals – pyritic, collophane-cemented, glauconite-cemented, gypsiferous, anhydrite-cemented, baritic, feldspar-cemented, etc.

The degree of cementation of a soil is determined qualitatively by utilizing finger pressure on the soil in one of the sample liners to disrupt the gross soil fabric. The three cementation descriptors are as follows:

- Weak – friable; crumbles or breaks with handling or slight finger pressure
- Moderate – friable; crumbles or breaks with considerable finger pressure
- Strong – not friable; will not crumble or break with finger pressure

8.1.5.3 *Structure*

This variable is used to qualitatively describe physical characteristics of soil that are important to incorporate into hydrogeological and/or geotechnical descriptions of soil at a site. Appropriate soil structure descriptors are as follows:

- Granular – spherically shaped aggregates with faces that do not accommodate adjoining faces
- Stratified – alternating layers of varying material or color with layers at least 6 mm (1/4 inch) thick; note thickness
- Laminated – alternating layers of varying material or color with layers less than 6 mm (1/4 inch) thick; note thickness
- Blocky – cohesive soil that can be broken down into small angular or subangular lumps that resist further breakdown
- Lensed – inclusion of a small pocket of different soil, such as small lenses of sand, should be described as homogeneous if it is not stratified, laminated, fissured, or blocky. If lenses of different soil are present, the soil being described can be termed homogeneous if the description of the lenses is included
- Prismatic or Columnar – particles arranged about a vertical line, ped is bounded by planar, vertical faces that accommodate adjoining faces; prismatic has a flat top; columnar has a rounded top
- Platy – particles are arranged about a horizontal plane

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

8.1.5.4 Other Features

- Mottled – soil that appears to consist of material of two or more colors in blotchy distribution
- Fissured – breaks along definite planes of fracture with little resistance to fracturing (determined by applying moderate pressure to sample using thumb and index finger)
- Slickensided – fracture planes appear polished or glossy, sometimes striated (parallel grooves or scratches)

8.1.6 **Development of Soil Description**

Develop standard soil descriptions according to the following examples. There are three principal categories under which all soil can be classified. They are described below.

8.1.6.1 Coarse-grained Soil

Coarse-grained soil is divided into sands and gravels. A soil is classified as a sand if over 50 percent of the coarse fraction is "sand-sized." It is classified as a gravel if over 50 percent of the coarse fraction is composed of "gravel-sized" particles.

The written description of a coarse-grained soil shall contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); grain size of coarse fraction; Munsell color and color number; moisture content; relative density; sorting; angularity; other features, such as stratification (sedimentary structures) and cementation, possible formational name, primary USCS classification, secondary USCS classification (when necessary), and approximate percentages of minor constituents (i.e., sand, gravel, shell fragments, rip-up clasts) in parentheses.

Example: POORLY-SORTED SAND WITH SILT, medium- to coarse-grained, light olive gray, 5Y 6/2, saturated, loose, poorly sorted, subrounded clasts, SW/SM (minor silt with approximately 20 percent coarse-grained sand-sized shell fragments, and 80 percent medium-grained quartz sand, and 5 percent to 15 percent ML).

8.1.6.2 Fine-grained Soil

Fine-grained soil is further subdivided into clays and silts according to its plasticity. Clays are rather plastic, while silts have little or no plasticity.

The written description of a fine-grained soil should contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); Munsell color; moisture content; consistency; plasticity; other features, such as stratification, possible formation name, primary USCS classification, secondary USCS classification (when necessary), and the percentage of minor constituents in parentheses.

Example: SANDY LEAN CLAY, dusky red, 2.5 YR 3/2, moist, firm, moderately plastic, thinly laminated, CL (70 percent fines, 30 percent sand, with minor amounts of disarticulated bivalves [about 5 percent]).

8.1.6.3 Organic Soil

For highly organic soil, describe the types of organic materials present as well as the type of soil constituents present using the methods described above. Identify the soil as an organic soil, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soil usually has a dark brown to black color and may have an organic odor. Often, organic soils will change color, (e.g., from black to brown) when exposed to air. Some organic soils will lighten in color significantly when air-dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

8.2 Example: ORGANIC CLAY, black, 2.5Y, 2.5/1, wet, soft, low plasticity, organic odor, OL (100 percent fines), weak reaction to HCl.

8.3 Rock Classification

The purpose of rock classification is to thoroughly describe the physical and mineralogical characteristics of a specimen and to classify it according to an established system. The generalized rock classification system described below was developed because, unlike the USCS for soils, there is no universally accepted rock classification system. In some instances, a more detailed and thorough rock classification system may be appropriate. Any modifications to this classification system, or the use of an alternate classification system should be considered during preparation of the site work plan. Both the CTO Manager and the QA Manager or Technical Director must approve any modifications to this classification system, or the use of another classification system.

Describing rock specimens on a common basis is essential so that rocks described by different site geologists are comparable. Site geologists describing rock specimens as a part of investigative activities must use the classification system described herein, or if necessary, another more detailed classification system. Use of a common classification system provides the most useful geologic database for all present and future subsurface investigations and remedial activities.

In order to provide a more consistent rock classification between geologists, a rock classification template has been designated as shown in **Error! Reference source not found.**. The template includes classification of rocks by origin and mineralogical composition. When classifying rocks, all site geologists shall use this template.

The site geologist shall describe the rock specimen and record the description in a boring log or logbook. The items essential for classification include (i.e., metamorphic foliated):

- Classification Name (i.e., schist)
- Color
- Mineralogical composition and percent
- Texture/Grain size (i.e., fine-grained, pegmatitic, aphlitic, glassy)
- Structure (i.e., foliated, fractured, lenticular)
- Rock Quality Designation (sum of all core pieces greater than two times the diameter of the core divided by the total length of the core run, expressed as a percentage)
- Classification symbol (i.e., MF)

Example: Metamorphic foliated schist: Olive gray, 5Y, 3/2, Garnet 25 percent, Quartz 45 percent, Chlorite 15 percent, Tourmaline 15 percent, Fine-grained with Pegmatite garnet, highly foliated, slightly wavy, MF.

9.0 Quality Control and Assurance

None

DEFINITION OF TERMS				
PRIMARY DIVISIONS			SYMBOLS	SECONDARY DIVISIONS
SEDIMENTARY ROCKS	Clastic Sediments	CONGLOMERATE	CG	Coarse-grained Clastic Sedimentary Rock types including: Conglomerates and Breccias
		SANDSTONE	SS	Clastic Sedimentary Rock types including: Sandstone, Arkose and Greywacke
		SHALE	SH	Fine-grained Clastic Sedimentary Rock types including: Shale, Siltstone, Mudstone and Claystone
	Chemical Precipitates	CARBONATES	LS	Chemical Precipitates including: Limestone, Crystalline Limestone, Fossiliferous Limestone Micrite and Dolomite
		EVAPORITES	EV	Evaporites including: Anhydrite, Gypsum, Halite, Travertine and Caliche
IGNEOUS ROCKS		EXTRUSIVE (Volcanic)	IE	Volcanic Rock types including: Basalt, Andesite, Rhyolite, Volcanic Tuff, and Volcanic Breccia
		INTRUSIVE (Plutonic)	II	Plutonic Rock types including: Granite, Diorite and Gabbro
METAMORPHIC ROCKS		FOLIATED	MF	Foliated Rock types including: Slate, Phyllite, Schist and Gneiss
		NON-FOLIATED	MN	Non-foliated Rock types including: Metaconglomerate, Quartzite and Marble

Figure 8-4: Rock Classification System

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

10.0 Data and Records Management

10.1 Document soil classification information collected during soil sampling onto the field boring logs, field trench logs, and into the field notebook. Copies of this information shall be sent to the **CTO Manager** for the project files.

10.2 Field notes will be kept during coring activities in accordance with SOP 3-03 – Recordkeeping, Sample Labeling, and Chain of Custody. The information pertinent to soil classification activities includes chronology of events, sample locations (x,y,z), time/date, sampler name, methods (including type of core liner/barrel, if applicable), sampler penetration and acceptability, sample observations, and the times and type of equipment decontamination. Deviations to the procedures detailed in the SOP should be recorded in the field logbook.

11.0 Attachments or References

American Society for Testing and Materials (ASTM). 2000. *Standard Practice for Description and Identification of Soils (Visual, Manual Procedure)*. D 2488-00. West Conshohocken, PA.

Birkeland, Peter W. 1984. *Soils and Geomorphology.* 3rd ed. New York: Oxford University Press.

Compton, Robert R. 1985. *Geology in the Field*. Hoboken, NJ: John Wiley & Sons, Inc.

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of the Navy (DON). 2007. *Navy Environmental and Natural Resources Program Manual*. OPNAV Instruction 5090.1c. October.

Folk, Robert L. 1980. *Petrology of Sedimentary Rocks*. Austin, TX: Hemphill Publishing Company.

Huang, Walter T. 1962. *Petrology*. New York: McGraw-Hill Book Company.

McCarthy, David F. 2005. *Essentials of Soil Mechanics and Foundations: Basic Geotechnics*. 7th Ed. Indianapolis, IN: Prentice Hall. July.

Munsell Color Company (Munsell). 2000. *Munsell Soil Color Chart*, (Revised). Baltimore.

Pettijohn, F.J. 1957. *Sedimentary Rocks*. 2nd Edition. New York: Harper and Brothers.

Rahn, Perry H. 1996. *Engineering Geology*. 2nd Edition. Indianapolis, IN: Prentice Hall. August

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Field Analysis of Ferrous Iron Using the HACH DR890 Colorimeter and HACH Method 8146

Procedure 3-18

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) is applicable for the field analysis of water, wastewater, and seawater for ferrous iron (Fe^{+2}). Concentrations ranging up to 3.00 mg/L can be analyzed, with higher concentrations analyzed by diluting the samples. The estimated detection limit is 0.03 mg/L. The presence of ferrous iron is indicative of a reducing state, therefore measurement of ferrous iron is useful in determining whether site conditions are reducing.

1.2 The 1,10-phenanthroline indicator in the AccuVac ampul reacts with ferrous iron (Fe^{+2}) in the sample to form an orange color in proportion to the iron concentration. Ferric iron (Fe+3) does not react. The ferric ion concentration can be determined by subtracting the ferrous iron concentration from results of a total iron test.

1.3 Samples are analyzed immediately after collection because ferrous iron readily oxidizes to ferric iron upon exposure to air. A minimum volume of approximately 100 mL of sample is required to complete this analysis. A HACH "AccuVac" ampul (a vacuum-sealed glass ampul containing 1,10-phenanthroline reagent) is broken, tip down in the sample, and allowed to react for the required three minutes. The colorimeter is zeroed and the sample analyzed. The instrument must be zeroed with a sample water blank prior to each analysis.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) Work Plan (WP) and/or direction from the Site Safety Officer (SSO).

2.2 Caution should be taken when working with all chemicals. Refer to the Material Safety Data Sheets (MSDSs) for the chemicals to be used.

2.3 Caution should be taken by the analyst with the sharp, broken glass that results after activating the AccuVac ampul.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Ferrous iron (Fe^{+2}) oxidizes into ferric iron (Fe^{+3}) rapidly on exposure to air and addition of oxidants. Samples must be analyzed immediately after sample collection.

4.2 Color development due to the reaction of ferrous iron with 1,10-phenanthroline is time dependent. Therefore, it is critical that the reaction time allowed between addition of the reagent from the AccuVac vial and color measurement be consistent for all analyses and carefully timed.

4.3 Care should be taken to avoid cross-contamination between field samples. The analyst should change gloves each time before handling the field sample for analysis. Sample color and turbidity will interfere

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

with colorimetric measurement. The colorimeter is therefore blank corrected with sample water prior to sample analysis and measurement.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The CTO Manager is responsible for ensuring that field analysis activities comply with this procedure. The CTO Manager is responsible for ensuring that all personnel involved in the field analysis shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all field analysis is conducted according to this procedure.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

6.1 Equipment

- HACH DR/890 colorimeter;
- 3 HACH "AccuVac ampuls" (to allow for diluted analyses if required) per well;
- HACH colorimeter sample cell (for the blank) 10-20-25 mL, with cap;
- Plastic cups and lids;
- Graduated cylinders;
- Kimwipes®;
- Pipetter; and
- Deionized ultra-filtered water (DIUF) for dilutions.

7.0 Procedure

7.1 Sample Analysis Procedure

Approximately 100 mL of sample will be collected in a plastic cup. Some sample is poured into the HACH 10-20-25 mL sample cell as a blank, and the instrument is zeroed with the blank. An AccuVac ampul is broken into the remainder of the sample in the cup, and set to react for 3 minutes. The sample is then analyzed. The procedures are outlined below.

7.1.1 Turn on the colorimeter by pressing EXIT. Press PRGM. When prompted, enter 33 and press ENTER.

7.1.2 Check historical ferrous iron concentrations for the sample location if available, and determine if sample dilution is necessary: If the anticipated concentration is above 3.00 mg/L, the upper end of the operating range of the colorimeter, a dilution is necessary. Prepare an appropriate dilution with DIUF using the graduated cylinder and pipette as needed. Do not use the volume indicators on the sample cell as these are not sufficiently accurate. Place the required volume of sample into the graduated cylinder, and fill to the desired volume with DIUF to the total volume. Swirl the cylinder by grasping the top with three fingers, and swinging in a circle three times. Record the volumes of sample and DIUF used for the dilution.

7.1.3 Pour at least 10 mL of the sample or diluted sample water into the 10-20-25 mL sample cell (not an AccuVac ampul). This will be used for the blank to zero the colorimeter.

7.1.4 Clean the sample cell with a damp Kimwipe, followed by a dry Kimwipe.

7.1.5 Place the 10-20-25 mL sample cell in the colorimeter, using the same orientation each time.

7.1.6 Cover, and press ZERO.

7.1.7 Pour at least 10mL of the unused sample or diluted sample into a plastic cup. Immerse an inverted AccuVac ampul in the sample water and snap off the tip on the bottom of the cup. Hold the ampul in place until water is no longer entering the ampul (about 4 seconds).

7.1.8 Remove the ampul from the cup, and quickly invert several times to mix. It is not necessary to block the top of the ampul as the liquid will not leak out.

7.1.9 Press TIMER, and the press ENTER on the colorimeter. A three minute reaction period will begin. During this reaction period, wipe off the AccuVac ampul with a Kimwipe.

7.1.10 When the 3-minute timer is up (colorimeter will beep), put the ampul in the colorimeter, cover tightly with the colorimeter cap, press READ, and record the reading.

7.1.11 Record the colorimeter reading on the sample worksheet.

7.1.12 If the reading is >3.00, collect more sample, dilute the sample, and repeat steps 4 through 12. If the sample was diluted and the reading is <0.03, collect more sample, prepare at a lower dilution, and reanalyze.

8.0 Quality Control and Assurance

8.1 Precision, Accuracy, and Contamination

8.1.1 Precision and sample variability is assessed through analysis of 1 field duplicate sample per day, or per 20 field samples, whichever is more frequent. Precision of 30% RPD is considered acceptable for field duplicate analyses. Note that the rapid oxidation of ferrous iron precludes replicate analysis of a sample.

8.1.2 Potential contamination is assessed through the analysis of blanks. The colorimeter must be zeroed with a sample water blank prior to analysis of each sample. For diluted sample analyses, the blank should consist of the diluted sample water at the same dilution as the sample.

8.2 Sample Collection, Preservation, and Storage

8.2.1 Sample preservation and storage is not possible since ferrous iron oxidizes rapidly. Samples should be analyzed immediately after collection.

8.2.2 Use a clean, unused plastic jar to transfer sample.

8.3 Method Performance

The HACH DR/890 colorimeter has an estimated detection limit of 0.03 mg/l Fe^{+2} and an analytical standard deviation (single operator) of 0.009 mg/l Fe^{+2}.

8.4 Pollution Prevention

All dilutions will be carefully recorded and tabulated for use in future site analyses to minimize the number of dilutions and analyses required.

8.5 Waste Management

8.5.1 Unused sample must be disposed of as per the sampling and analysis plan, quality assurance project plan, and/or work plan.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

8.5.2 The reacted AccuVac ampul should be placed in the AccuVac Vial Destruct Unit (AVDU) (a 1-L HDPE sample bottle with a large rock), and shaken to break the ampul. Replace the lid of the AVDU with the lid that has holes punctured in the top, and drain all remaining liquid from the AVDU into a container with the sample water for drumming, disposal, etc. Dispose of the glass-filled AVDU as unregulated solid waste.

8.5.3 Liquids drained from the test kits should be diluted five-fold with tap water for discharge to drain, or diluted with purge water and drummed with the purge water for disposal according to the sampling and analysis plan and/or work plan.

9.0 Records, Data Analysis, Calculations

9.1 Results should be reviewed prior to leaving the field to be sure field duplicates were within acceptance range and results did not exceed the instrument's range (3.00 mg/l).

9.2 The dilution factor is calculated as follows:

9.3 DF = (Volume of DIUF water + Volume of sample water) ÷ Volume of sample water

9.4 Concentration of ferrous iron in sample water = reading on colorimeter x DF

9.5 Example: If 10 mL of sample are diluted with 90 mL of DIUF water and the colorimeter reading was 2.3 mg/L, then:

9.6 Ferrous Iron Concentration (mg/L) = 2.3 mg/L x 10 = 23 mg/L

9.7 Unanticipated changes to the procedures or materials described in this POP (deviations) will be appropriately documented in the project records.

9.8 Records associated with the activities described in this POP will be maintained according to the document management policy for the project.

10.0 Attachments or References

10.1 United States Environmental Protection Agency. 2001. Guidance for Preparing Standard Operating Procedures (SOPs). EPA QA/G-6. EPA/240/B-01/004. USEPA Office of Environmental Information, Washington, DC. March 2001.

10.2 *Hach DR/890 Colorimeter Procedures Manual*, Edition 7, HACH Company, December 2005.

10.3 *Hach DR/820, DR/850, DR890 Portable Datalogging Colorimeter Instrument Manual*, Revision 5, HACH Company, 1997 1999.

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Headspace Screening for Total VOCs

Procedure 3-19

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the basic techniques for using headspace analysis to screen for volatile organics in contaminated soils using a portable Photo Ionization Detector (PID) or Flame Ionization Detector (FID).

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**. Note that headspace screening usually requires Level D personal protection unless there is a potential for airborne exposure to site contaminants. Under circumstances where potential airborne exposure is possible respiratory protective equipment may be required based on personal air monitoring results. Upgrades to Level C will be coordinated with the Site Safety Officer (SSO) or **CTO Manager**.

2.2 Health and safety hazards and corresponding precautions include, but are not limited to, the following:

2.2.1 Dermal contact with contaminated soil. Personnel should treat all soil as potentially contaminated and wear chemically impervious gloves. Minimize skin contact with soil by using sampling instruments such as stainless steel spades or spoons. Do not touch any exposed skin with contaminated gloves.

2.2.2 Inhalation hazards. Appropriate air monitoring should be conducted to ensure that organic vapor concentrations in the breathing zone do not exceed action levels as specified in the Site-Specific HASP. When ambient temperatures are low enough to require warming samples using the vehicle heater, the vehicle's windows should be opened enough to prevent the build-up of any organic vapors. Use the PID or FID to verify the airborne concentrations in the vehicle remain below applicable action levels. Note that many volatile organic compounds (VOCs) are flammable and all precautions must be observed to eliminate any potential ignition sources.

2.2.3 Shipping limitations. Follow applicable regulations when shipping FID/PID equipment. When shipping an FID by air, the hydrogen tank must be bled dry. Calibration gas canisters are considered dangerous goods and must be shipped according to IATA and DOT regulations. Consult your EHS Coordinator and check with your shipping company to determine the correct shipping procedures

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Regardless of which gas is used for calibration, the instrument will respond to all analytes present in the sample that can be detected by the type of lamp used in the PID.

4.2 Moisture will generate a positive interference in the concentration measured for a PID and is characterized by a slow increase in the reading as the measurement is made. Care must be taken to

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

minimize uptake of moisture to the extent possible. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.3 Uptake of soil into the PID must be avoided as it will compromise instrument performance by blocking the probe, causing a positive interference, or dirtying the PID lamp. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.4 The user should listen to the pitch of the sampling pump. Any changes in pitch may indicate a blockage and corrective action should be initiated.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The CTO Manager is responsible for ensuring that the collection of headspace readings comply with this procedure. The CTO Manager is responsible for ensuring that all personnel involved in the collection of headspace readings shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all headspace readings are conducted according to this procedure as well as verifying that the PID/FID is in proper operating condition prior to use and for implementing the calibration.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

6.1 The following materials must be on hand in good operating condition and/or in sufficient quantity to ensure that proper field analysis procedures may be followed:

- Calibrated PID/FID instrument;
- Top-sealing "Zip-Loc" type plastic bags – or – 16 ounces of soil or "mason-" type glass jars and aluminum foil;
- Project field book and/or boring logs;
- Personal Protective Equipment (PPE) as specified in the project HASP; and
- Material Safety Data Sheets (MSDSs) for any chemicals or site-specific contaminants.

7.0 Procedure

7.1 **Preparation**

Review available project information to determine the types of organic vapors that will likely be encountered to select the right instrument. The two basic types of instruments are FIDs and PIDs.

FIDs work well with organic compounds that have relatively lightweight molecules, but may have problems detecting halogenated compounds or heavier organic compounds; FIDs can detect methane for example. Since the FID uses a flame to measure organic compounds, ensure that work is conducted in an atmosphere, which is free of combustible vapors. If ambient temperatures are below 40°F, the flame of the FID may be difficult to light.

When using a PID, select an instrument that can measure the ionization potential of the anticipated contaminants of concern. PIDs work well with a range of organic compounds and can detect some halogenated hydrocarbons; PIDs cannot detect methane. The correct ultraviolet (UV) light bulb must be selected according to the types of organic vapors that will likely be encountered. The energy of the UV light must equal or exceed the ionization potential of the organic molecules that the PID will measure. The NIOSH Pocket Guide to Chemical Hazards is one source for determining ionization potentials for different chemicals. Bulbs available for PIDs include 9.4 eV, 10.6 (or 10.2) eV, and 11.7 eV bulbs. The 10.6 eV bulb is most commonly used as it detects a fairly large range of organic molecules and does not burn out as easily as the 11.7 eV bulb. The 9.4 eV bulb is the most rugged, but detects only a limited range of compounds. Under very humid or very cold ambient conditions, the window covering the UV light may fog up, causing inaccurate readings. Ask the **SSO** about correction factors when high humidity conditions exist.

After selecting the correct instrument, calibrate the PID/FID according to the manufacturer's instructions. Record background/ambient levels of organic vapors measured on the PID/FID after calibration and make sure to subtract the background concentration (if any) from your readings. Check the PID/FID readings against the calibration standard every 20 readings or at any time when readings are suspected to be inaccurate, and recalibrate, if necessary. Be aware that, after measuring highly contaminated soil samples, the PID/FID may give artificially high readings for a time.

7.2 Top-Sealing Plastic Bag

Place a quantity of soil in a top-sealing plastic bag and seal the bag immediately. The volume of soil to be used should be determined by the **CTO Manager** or **Field Manager**. The volume of soil may vary between projects but should be consistent for all samples collected for one project. Ideally, the bag should be at least 1/10th-filled with soil and no more than half-filled with soil. Once the bag is sealed, shake the bag to distribute the soil evenly. If the soil is hard or clumpy, use your fingers to gently work the soil (through the bag) to break up the clumps. Do not use a sampling instrument or a rock hammer since this may create small holes in the plastic bag and allow organic vapors to escape. Alternatively, the sample may be broken up before it is placed in the bag. Use a permanent marker to record the following information on the outside of the bag:

- Site identification information (i.e., borehole number);
- Depth interval; and
- Time the sample was collected. For example: "SS-12, 2-4 ft, @1425".

Headspace should be allowed to develop before organic vapors are measured with a PID/FID. The amount of time required for sufficient headspace development will be determined by the project-specific sampling plan and the ambient temperature. Equilibration time should be the same for all samples to allow an accurate comparison of organic vapor levels between samples. However, adjustments to equilibration times may be necessary when there are large variations in ambient temperature from day to day. When ambient temperatures are below 32°F, headspace development should be within a heated building or vehicle. When heating samples, be sure there is adequate ventilation to prevent the build-up or organic vapors above action levels.

Following headspace development, open a small opening in the seal of the plastic bag. Insert the probe of a PID/FID and seal the bag back up around the probe as tightly as possible. Alternatively, the probe can be inserted through the bag to avoid loss of volatiles. Since PIDs and FIDs are sensitive to moisture, avoid touching the probe to the soil or any condensation that has accumulated inside of the bag. Since the PID/FID consumes organic vapors, gently agitate the soil sample during the reading to release fresh organic vapors from the sample. Erratic meter response may occur at high organic vapor concentrations or conditions of elevated headspace moisture, in which case, headspace data should be discounted. Record the highest reading on the field form or in the field notebook as described in Section 9.

7.3 **Jar and Aluminum Foil (Alternate Method)**

Half-fill a clean glass jar with the soil sample to be screened. Quickly cover the jar's opening with one to two sheets of clean aluminum foil and apply the screw cap to tightly seal the jar. Allow headspace development for at least ten minutes. Vigorously shake the jar for 15 seconds, both at the beginning and at the end of the headspace development period. Where ambient temperatures are below 32°F (0°C), headspace development should be within a heated area. When heating samples, be sure there is adequate ventilation to prevent the build-up of organic vapors above action levels.

Subsequent to headspace development, remove the jar lid and expose the foil seal. Quickly puncture the foil seal with the instrument sampling probe, to a point about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particulates. As an alternative, use a syringe to withdraw a headspace sample, and then inject the sample into the instrument probe or septum-fitted inlet. This method is acceptable contingent upon verification of methodology accuracy using a test gas standard. Following probe insertion through the foil seal or sample injection to probe, record the highest meter response on the field form or in the field notebook. Using foil seal/probe insertion method, maximum response should occur between two and five seconds. Erratic meter response may occur at high organic vapor concentrations or conditions of elevated headspace moisture, in which case, headspace data should be discounted.

8.0 Quality Control and Assurance

Quality Assurance/Quality Control (QA/QC) will include the collection of duplicate samples. In general, one duplicate will be collected per 20 samples. Organic vapor concentrations measured in the primary and duplicate samples should be similar within plus or minus 20 percent. The frequency of headspace duplicate collection will be determined by the project manager/task manager. The PID/FID instrument must be calibrated according to the manufacturer's instructions before beginning screening, and checked or recalibrated every 20 analyses or when readings are suspected to be inaccurate. Record ambient organic vapor levels in the field notebook and on the field form. Periodically check ambient organic vapor levels. If ambient levels have changed more than 20 percent, recalibrate the PID/FID. Make sure readings are not collected near a vehicle exhaust or downwind of a drill rig exhaust. If grossly contaminated soil is encountered, decontaminate sampling instruments between samples and/or change contaminated gloves to avoid cross contaminating less contaminated samples.

9.0 Records, Data Analysis, Calculations

9.1 All data generated (results and duplicate comparisons) will be recorded in the field notebook and/or on the field form. Any deviation from the outlined procedure will also be noted. Field conditions (ambient temperature, wind, etc.) should also be recorded in the field notebook.

9.2 Readings may be recorded in a field notebook, on a boring log, or on an appropriate form specific to the project. The form should include the following information:

- When the PID/FID was calibrated (date/time) and calibration standard used;
- Background/ambient concentrations measured after PID/FID calibration;
- Location of sample (i.e., bore-hole number);
- Depth interval of sample measured;
- Lithology of material measured; and
- PID/FID reading and units of measure.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

9.3 Note that if PID/FID measurements are recorded on a boring log, it is not necessary to duplicate information in the column where the PID/FID readings are recorded (e.g., borehole number, depth interval, lithology type).

9.4 All documentation will be stored in the project files and retained following completion of the project.

10.0 Attachments or References

SOP 3-20 Operation and Calibration of a Photoionization Detector

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Operation and Calibration of a Photoionization Detector

Procedure 3-20

1.0 Purpose and Scope

1.1 Purpose and Applicability

1.1.1 This standard operating procedure (SOP) describes the procedures that will be followed by field staff for operation and calibration of a photoionization detector (PID). The PID is primarily used by AECOM personnel for safety and survey monitoring of ambient air, determining the presence of volatiles in soil and water, and detecting leakage of volatiles.

1.1.2 PIDs routinely used by field personnel include the Photovac Microtip, Thermoelectron 580EZ, and MiniRAE 2000. Personnel responsible for using the PID should first read and thoroughly familiarize themselves with the instrument instruction manual.

1.2 Principle of Operation

1.2.1 The PID is a non-specific vapor/gas detector. The unit generally consists of a hand-held probe that houses a PID, consisting of an ultraviolet (UV) lamp, two electrodes, and a small fan which pulls ambient air into the probe inlet tube. The probe is connected to a readout/control box that consists of electronic control circuits, a readout display, and the system battery. Units are available with UV lamps having an energy from 9.5 electron volts (eV) to 11.7 eV.

1.2.2 The PID analyzer measures the concentration of trace gas present in the atmosphere by photoionization. Photoionization occurs when an atom or molecule absorbs a photon of sufficient energy to release an electron and become a positive ion. This will occur when the ionization potential of the molecule (in electron volts (eV)) is less than the energy of the photon. The source of photons is an ultraviolet lamp in the probe unit. Lamps are available with energies ranging from 9.5 eV to 11.7 eV. All organic and inorganic vapor/gas compounds having ionization potentials lower than the energy output of the UV lamp are ionized and the resulting potentiometric change is seen as a positive reading on the unit. The reading is proportional to the concentration of organics and/or inorganics in the vapor.

1.2.3 Sample gases enter the probe through the inlet tube and enter the ion chamber where they are exposed to the photons emanating from the UV lamp. Ionization occurs for those molecules having ionization potentials near to or less than that of the lamp. A positive- biased polarizing electrode causes these positive ions to travel to a collector electrode in the chamber. Thus the ions create an electrical current which is amplified and displayed on the meter. This current is proportional to the concentration of trace gas present in the ion chamber and to the sensitivity of that gas to photoionization.

1.2.4 In service, the analyzer is first calibrated with a gas of known composition equal to, close to, or representative of that to be measured. Gases with ionization potentials near to or less than the energy of the lamp will be ionized. These gases will thus be detected and measured by the analyzer. Gases with ionization potentials greater than the energy of the lamp will not be detected. The ionization potentials of the major components of air, i.e., oxygen, nitrogen, and carbon dioxide, range from about 12.0 eV to 15.6 eV and are not ionized by any of the lamps available. Gases with ionization potentials near to or slightly higher than the lamp are partially ionized, with low sensitivity.

1.3 Specifications

1.3.1 Refer to the manufacturer's instructions for the technical specifications of the instrument being used. The operating concentration range is typically 0.1 to 2,000 ppm isobutylene equivalent.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**.

2.2 Only PIDs stamped Division I Class I may be used in explosive atmospheres. Refer to the project HASP for instructions pertaining to instrument use in explosive atmospheres.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Regardless of which gas is used for calibration, the instrument will respond to all analytes present in the sample that can be detected by the type of lamp used in the PID.

4.2 Moisture will generate a positive interference in the concentration measured for a PID and is characterized by a slow increase in the reading as the measurement is made. Care must be taken to minimize uptake of moisture to the extent possible. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.3 Uptake of soil into the PID must be avoided as it will compromise instrument performance by blocking the probe, causing a positive interference, or dirtying the PID lamp. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.4 The user should listen to the pitch of the sampling pump. Any changes in pitch may indicate a blockage and corrective action should be initiated.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The CTO Manager is responsible for ensuring that the operation and calibration activities comply with this procedure. The CTO Manager is responsible for ensuring that all personnel involved in the operation and calibration shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all operation and calibration activities are conducted according to this procedure.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

- Calibration Gas: Compressed gas cylinder of isobutylene in air or similar stable gas mixture of known concentration. The selected gas should have an ionization potential similar to that of the vapors to be monitored, if known. The concentration should be at 50-75% of the range in which the instrument is to be calibrated;

- Regulator for calibration gas cylinder;
- Approximately 6 inches of Teflon® tubing;
- Tedlar bag (optional);
- Commercially-supplied zero grade air (optional);
- "Magic Marker" or "Sharpie" or other waterproof marker;
- Battery charger;
- Moisture traps;
- Spare lamps;
- Manufacturer's instructions; and
- Field data sheets or logbook/pen.

7.0 Procedure

7.1 Preliminary Steps

7.1.1 Preliminary steps (battery charging, check-out, calibration, maintenance) should be conducted in a controlled or non-hazardous environment.

7.2 Calibration

7.2.1 The PID must be calibrated in order to display concentrations in units equivalent to ppm. First a supply of zero air (ambient air or from a supplied source), containing no ionizable gases or vapors is used to set the zero point. A span gas, containing a known concentration of a photoionizable gas or vapor, is then used to set the sensitivity.

7.2.2 Calibrate the instrument according to the manufacturer's instructions. Record the instrument model and identification number, the initial and adjusted meter readings, the calibration gas composition and concentration, and the date and the time in the field records.

7.2.3 If the calibration cannot be achieved or if the span setting resulting from calibration is 0.0, then the lamp must be cleaned (Section 7.4).

7.3 Operation

7.3.1 Turn on the unit and allow it to warm up (minimum of 5 minutes). Check to see if the intake fan is functioning; if so, the probe will vibrate slightly and a distinct sound will be audible when holding the probe casing next to the ear. Also, verify on the readout display that the UV lamp is lit.

7.3.2 Calibrate the instrument as described in Section 7.2, following the manufacturer's instructions. Record the calibration information in the field records.

7.3.3 The instrument is now operational. Readings should be recorded in the field records.

7.3.4 When the PID is not being used or between monitoring intervals, the unit may be switched off to conserve battery power and UV lamp life; however, a "bump" test should be performed each time the unit is turned on and prior to taking additional measurements. To perform a bump test, connect the outlet tubing from a Tedlar bag containing a small amount of span gas to the inlet tubing on the unit and record the reading. If the reading is not within the tolerance specified in the project plan, the unit must be recalibrated.

7.3.5 At the end of each day, recheck the calibration. The check will follow the same procedures as the initial calibration (Section 7.2) except that no adjustment will be made to the instrument. Record the information in the field records.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

7.3.6 Recharge the battery after each use (Section 7.4).

7.3.7 When transporting, ensure that the instrument is packed in its stored condition in order to prevent damage.

7.4 **Routine Maintenance**

7.4.1 Routine maintenance associated with the use of the PID includes charging the battery, cleaning the lamp window, replacing the detector UV lamp, replacing the inlet filter, and replacing the sample pump. Refer to the manufacturer's instructions for procedures and frequency.

7.4.2 All routine maintenance should be performed in a non-hazardous environment.

7.5 **Troubleshooting Tips**

7.5.1 One convenient method for periodically confirming instrument response is to hold the sensor probe next to the tip of a magic marker. A significant reading should readily be observed.

7.5.2 Air currents or drafts in the vicinity of the probe tip may cause fluctuations in readings.

7.5.3 A fogged or dirty lamp, due to operation in a humid or dusty environment, may cause erratic or fluctuating readings. The PID should never be operated without the moisture trap in place.

7.5.4 Moving the instrument from a cool or air-conditioned area to a warmer area may cause moisture to condense on the UV lamp and produce unstable readings.

7.5.5 A zero reading on the meter should not necessarily be interpreted as an absence of air contaminants. The detection capabilities of the PID are limited to those compounds that will be ionized by the particular probe used.

7.5.6 Many volatile compounds have a low odor threshold. A lack of meter response in the presence of odors does not necessarily indicate instrument failure.

7.5.7 When high vapor concentrations enter the ionization chamber in the PID the unit can become saturated or "flooded". Remove the unit to a fresh air environment to allow the vapors to be completely ionized and purged from the unit.

8.0 Quality Control and Assurance

8.1 The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Sampling and Analysis Plan (SAP), hereafter referred to as the project plan.

8.2 Calibration of the PID will be conducted at the frequency specified in the project plan. In the absence of project-specific guidance, calibration will be performed at the beginning of each day of sampling and will be checked at the end of the sampling day or whenever instrument operation is suspect. The PID will sample a calibration gas of known concentration. The instrument must agree with the calibration gas within ±10%. If the instrument responds outside this tolerance, it must be recalibrated.

8.3 Checks of the instrument response (Section 7.5) should be conducted periodically and documented in the field records.

9.0 Records, Data Analysis, Calculations

Safety and survey monitoring with the PID will be documented in a bound field logbook, or on standardized forms, and retained in the project files. The following information is to be recorded:

- Project name and number;
- Instrument manufacturer, model, and identification number;

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

- Operator's signature;
- Date and time of operation;
- Calibration gas used;
- Calibration check at beginning and end of day (meter readings before adjustment);
- Span setting after calibration adjustment;
- Meter readings (monitoring data obtained);
- Instances of erratic or questionable meter readings and corrective actions taken; and
- Instrument checks and response verifications – e.g., battery check, magic marker response (Section 7.5) or similar test.

10.0 Attachments or References

United States Environmental Protection Agency. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM). USEPA, Region 4, SESD, Enforcement and Investigations Branch, Athens, GA. November 2001.

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Surface and Subsurface Soil Sampling Procedures

Procedure 3-21

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the procedures for soil sampling. The procedure includes surface and subsurface sampling by various methods using hand auguring, test pit, direct-push, and split-spoon equipment.

1.2 The procedure includes soil sampling for volatile organic compounds (VOCs). For project specific information (e.g. sampling depths, equipment to be used, and frequency of sampling), refer to the Sampling and Analysis Plan (SAP), which takes precedence over these procedures. Surface soil sampling, typically considered to be up to two feet below ground surface by EPA standards, is typically accomplished using hand tools such as shovels or hand augers. Test pit samples are considered subsurface samples, although normally collected via hand tools similar to surface soil sampling or by excavation machinery. Direct-push and split-spoon sampling offer the benefit of collecting soil samples from a discrete or isolated subsurface interval, without the need of extracting excess material above the target depth. These methods dramatically reduce time and cost associated with disposal of material from soil cuttings when compared to test pit sampling. In addition, direct-push and split-spoon sampling methods can obtain samples at targeted intervals greater than 15 feet in depth, allowing for discrete depth soil sampling while speeding up the sampling process. Direct-push methods work best in medium to fine-grained cohesive materials such as medium to fine sands, silts, and silty clay soils. Split-spoon sampling works well in all types of soil, but is somewhat slower than direct-push methods. Samples are composited so that each sample contains a homogenized representative portion of the sample interval. Due to potential loss of analytes, samples for volatile analysis are not composited. Samples for chemical analysis can be collected by any of the above-mentioned sampling methods, as disturbed soil samples. Undisturbed samples are collected, sealed, and sent directly to the laboratory for analysis. For undisturbed samples, the samples are not homogenized.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**.

2.2 Before soil sampling commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated soil sampling locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Low recovery of soil from sampling equipment will prevent an adequate representation of the soil profile and sufficient amount of soil sample. If low recovery is a problem, the hole may be offset and re-advanced, terminated, or continued using a larger diameter sampler.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

4.2 Asphalt in soil samples can cause false positive results for hydrocarbons. To ensure samples are free of asphalt, do not collect samples that may contain asphalt. If the collection of samples potentially containing asphalt is unavoidable, note the sampling depths at which the presence of asphalt are suspected.

4.3 Instrumentation interferences addressed in SOPs for Calibration of the Photoionization Detector (PID), Headspace Screening for Total Volatile Organics, and Equipment Decontamination must also be considered.

4.4 Cross contamination from sampling equipment must be prevented by using sampling equipment constructed of stainless steel that is adequately decontaminated between samples.

5.0 Training and Qualifications

5.1 **Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The CTO Manager is responsible for ensuring that soil sampling activities comply with this procedure. The CTO Manager is responsible for ensuring that all personnel involved in soil sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all soil sampling activities are conducted according to this procedure.

5.2.4 All Field Personnel are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

The depth at which samples will be collected and the anticipated method of sample collection (direct-push, split-spoon, hand auger, shovel, or test pits) will be presented in the SAP. The following details equipment typically needed for soil sampling, based on the various methods. See the SAP for specific detail of equipment and supply needs.

6.1 Depending on the nature of suspected contamination, field screening instrumentation may be used for direct sampling. Appropriate instrumentation and calibration standards should be available. If volatile organic contaminants are suspected and a PID will be used, refer to the equipment and instrumentation listed in SOP 3-20 Operation and Calibration of a Photoionization Detector. Equipment in this SOP includes but is not limited to:

- PID/FID;
- Calibration gas; and
- Tedlar® gas bags (for calibration).

6.2 If field screening methods include jar headspace screening for volatile organics, refer to the equipment and procedure in SOP 3-19 Headspace Screening for Total VOCs. Equipment in this SOP includes but is not limited to:

- Clean soil ("drillers jars") jars; and
- Aluminium foil.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

6.3 Appropriate decontamination procedures must be followed for sampling equipment. Refer to SOP 3-06 Equipment Decontamination. Equipment in this SOP includes but is not limited to:

- Phosphate-free detergent;
- Isopropyl Alcohol;
- Tap water;
- Deionized Ultra-Filtered (DIUF) Water;
- Plastic buckets or washbasins;
- Brushes; and
- Polyethylene sheeting.

6.4 The following general equipment is needed for all soil sampling, regardless of method:

- Stainless steel bowls;
- Stainless steel trowels;
- Appropriate sample containers for laboratory analysis;
- Personal Protective Equipment (PPE);
- Logbook;
- Cooler and ice for preservation; and
- Stakes and flagging to document sampling location.

6.5 The following additional equipment is needed for volatile organic sampling:

- Electronic pan scale and weights for calibration; and
- Syringes or other discrete soil core samplers.

6.6 The following additional equipment may be needed for surface and test pit soil sampling:

- Hand Auger

6.7 The following additional equipment may be needed for soil sampling from direct push and/or split-spoon equipment:

- Tape measure or folding carpenter's rule for recording the length of soil recovered.

Note: All subsurface drilling equipment will be provided and maintained by the subcontractor.

7.0 Procedure

7.1 **General Soil Sampling Procedure for All Soil Sampling Methods**

7.1.1 Record the weather conditions and other relevant on-site conditions.

7.1.2 Select the soil sampling location, clear vegetation if necessary, and record the sampling location identification number and pertinent location details.

7.1.3 Verify that the sampling equipment is properly decontaminated, in working order, and situated at the intended sampling location.

7.1.4 Place polyethylene sheeting on the ground and assemble all necessary sampling equipment on top of it. Cover surfaces onto which soils or sampling equipment will be placed (i.e. tables with polyethylene sheeting).

7.1.5 Follow the appropriate procedures listed below for either surface, split-spoon, direct push, or test pit sample collection (7.2, 7.3, 7.4, and 7.5 respectively).

7.1.6 Collect soil samples according to procedures listed in Section 7.6 depending on project specific analyses.

7.1.7 Record date/time, sample ID, and sample descriptions in the field logbook or field form. A sketch or description of the location may also be recorded so the sample location can be re-constructed, especially if the location will not be recorded using global positioning satellite (GPS) equipment.

7.1.8 Immediately label the sample containers and place them on ice, if required for preservation. Complete the chain-of-custody form(s) as soon as possible.

7.1.9 Dispose of all excess excavated soil in accordance with the SAP.

7.1.10 If required, mark the sample location with a clearly labelled wooden stake or pin flag. If the location is on a paved surface, the location may be marked with spray paint.

7.1.11 Decontaminate the sampling equipment according to SOP 3-06 Equipment Decontamination.

7.2 Surface Sampling

7.2.1 The criteria used for selecting surface soil locations for sampling may include the following:

- Visual observations (soil staining, fill materials);
- Other relevant soil characteristics;
- Site features;
- Screening results;
- Predetermined sampling approach (i.e. grid or random); and
- Sampling objectives as provided in the SAP.

7.2.2 The following procedures are to be used to collect surface soil samples. Surface soils are considered to be soils that are up to two feet below ground surface, though state regulations and project objectives may define surface soils differently; therefore, the SAP should be consulted for direction on the depth from which to collect the surface soil samples. Sampling and other pertinent data and information will be recorded in the field logbook and/or on field forms. Photographs may be taken as needed or as specified in the SAP.

1. Gently scrape any vegetative covering until soil is exposed. Completely remove any pavement.
2. Remove soil from the exposed sampling area with a trowel, hand auger, or shovel. Put soils within the sampling interval in a stainless steel bowl for homogenizing. Monitor the breathing zone and sampling area as required in the HASP.
3. For VOC analyses, collect representative soil samples directly from the recently-exposed soil using a syringe or other soil coring device (e.g., TerraCore®, EnCore®). Follow procedures in Section 7.6.1 for VOC sampling.
4. Collect sufficient soil to fill all remaining sample jars into a stainless steel bowl. Homogenize the soil samples to obtain a uniform soil composition which is representative of the total soil sample collected according to the following procedure:

    a) Remove all rocks and non-soil objects using a stainless steel spoon or scoop.

b) Form a cone shaped mound with the sample material, then flatten the cone and split the sample into quarters.

c) Use the stainless steel spoon/scoop to mix the quarter samples that are opposite.

d) After mixing the opposite quarters, reform the cone shaped mound.

e) Repeat this procedure a minimum of five (5) times, removing any non-soil objects and breaking apart any clumps.

7.3 Split-Spoon Sampling

7.3.1 At each boring location, the frequency and depth of split-spoon samples will be determined from the SAP. Split-spoon samples may be collected continuously, intermittently, or from predetermined depths.

7.3.2 Split-spoon samplers shall be driven into undisturbed soil by driving the spoon ahead of the drill augers/casing. In cohesive soils, or soils where the borehole remains open (does not collapse), two split-spoon samples may be taken prior to advancing the augers/casing.

7.3.3 After split-spoons are retrieved, open the split-spoon and measure the recovery of soil. If a PID will be used for screening, immediately scan the recovered sample for VOCs using the PID. Scan the recovered soil boring by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the highest PID reading and the depth at which it was observed along with all other pertinent observations. If required in the SAP, VOC and headspace samples should be collected (see Section 7.6.1) prior to logging the sample.

7.3.4 If headspace screening for VOCs is required in the SAP, collect a soil sample (as defined in the SAP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.3.5 Soils collected using the split-spoon sampler will be logged by the field representative using the procedure required in the SAP.

7.3.6 Collect the remainder of the sample volume required into a stainless steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.3.7 The SAP may specify that intervals to be sent to the laboratory be determined by visual observation and/or highest PID screening or headspace results, which can only be determined once the boring is complete. In this instance, a VOC sample should be collected at each interval. The remainder of the soil from that interval will be set aside in a clearly labelled stainless steel bowl covered with aluminium foil. Once the boring has been completed and the sample interval has been determined, the remainder of the soil can be homogenized according to Section 7.2 and submitted for laboratory analysis.

7.3.8 Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the SAP (e.g., completed as a monitoring well, backfilled with bentonite, etc).

7.4 Direct Push Sampling

At each boring location, the frequency of direct-push samples will be determined from the SAP. Typically, samples with direct-push equipment are collected in 4 foot (ft) intervals, but smaller (e.g., 2 ft) and larger (e.g., 5 ft) intervals are also possible.

1. Sample using Macro-Core samplers with acetate liners to obtain discrete soil samples at the depths specified in the SAP.

2. Cut open the acetate liner. If required in the SAP, immediately scan the recovered soil boring for VOCs using a PID by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

highest PID reading and the depth at which it was observed along with all other pertinent observations. VOC and headspace samples, if required in the SAP should be collected (see Section 7.6.1) prior to logging the sample.

3. If required in the SAP, collect a soil sample (as defined in the SAP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

4. Soils collected using the direct-push sampler will be logged by the by the field representative using the procedure required in the SAP.

5. Collect the remainder of the sample into a stainless steel bowl. Homogenize the soil collected so that the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

6. Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the SAP (e.g., completed as a monitoring well, backfilled with bentonite, etc).

7.5 Test Pit Sampling

7.5.1 Excavate the test pit to the desired depth.

7.5.2 Using the excavator bucket, collect soil samples as specified in the SAP. Collect a sample and perform screening analyses as required by the SAP. If VOCs contamination is suspected, perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.5.3 Collect the sample from center of the bucket to avoid potential contamination from the bucket.

7.5.4 VOC samples should also be collected from an undisturbed section soil in the excavator bucket. The top layer of exposed soil should be scraped away just prior to collecting the VOC samples.

7.5.5 Collect the remainder of the sample volume required into a stainless steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.5.6 Dispose of all excavated soil according to the SAP.

7.6 Sample Collection Methods

7.6.1 Volatile Organics Sampling

For soils collected for analyses of volatile organics, including Volatile Petroleum Hydrocarbons (VPH) or other purgable compounds, a closed system is maintained. From collection through analysis, the sample bottles are not opened. The bottle kit for a routine field sample for these analyses will typically include three 40-mL VOA vials and one soil jar. Two 40-mL VOA vials will contain either 5 mL reagent water or 5 mL sodium bisulfate and magnetic stir bars (i.e., low level vials). The third VOA vial will contain 15 mL methanol with no magnetic stir bar (i.e., high level vial). These vials are usually provided by the laboratory and are pre-weighed, with the tare weight recorded on the affixed sample label. No additional sample labels are affixed to the VOA vials, as addition of a label would alter the vial weight. All information is recorded directly on the sample label using an indelible marker. The soil jar is provided for percent solids determination. For VOC or VPH analyses, samples are collected prior to sample homogenization. Collect the VOC sample in accordance with the procedure described below.

1. Determine the soil volume necessary for the required sample weight, typically 5 grams:

   a) Prepare a 5 mL sampling corer (e.g., Terra Core®) or cut-off plastic syringe.

   b) Tare the sampler by placing it on the scale, and zeroing the scale.

   c) Draw back the plunger to the 5 gram mark or 5mL (5cc) mark on cut-off syringe, and insert the open end of the sampler into an undisturbed area of soil with a twisting motion, filling the

sampler with soil. Note the location of the plunger with respect to the milliliter (cc) or other graduation printed on the sampler.

d) Weigh the filled sampler, and remove or add soil until the desired weight is obtained. Note the location of the plunger which corresponds to this weight. Do not use this sample for laboratory analysis.

2. Once the required soil volume has been determined, pull the plunger back to this mark and hold it there while filling the syringe for each sample.

3. Collect 5 grams of soil using the cut-off syringe or Terra Core® sample device. Extrude the 5-grams of soil into one of the low level 40-mL VOA vials. Quickly wipe any soil from the threads of the VOA vial with a clean Kimwipe® and immediately close the vial. It is imperative that the threads be free from soil or other debris prior to replacing the cap on the vial in order to maintain the closed system necessary for the analysis.

4. Gently swirl the vial so that all of the soil is fully wetted with the preservative.

5. Fill the other low level 40 mL VOA vial in this manner.

6. Repeat the process for the high level VOA vials, only for the high level VOA vial three 5 gram aliquots (i.e., 15 grams total) should be extruded into the high level VOA vial.

   NOTE: Depending on the laboratory, some high level VOA vials only contain 5 mL or 10 mL of methanol. If this is the case, either 5 grams total or 10 grams total, respectively, should be extruded into the high level VOA vial. In other words, the mass of soil in grams should be identical to the volume of methanol in mL (i.e., 1:1 ratio of soil to methanol).

7. Collect any additional QC sample collected (e.g., field duplicate, MS, and MSD) in the same manner as above.

8. Fill the 4-oz glass jar with soil from the same area for percent moisture determination.

7.6.2 Soil Sampling Method (All other analyses except VOC/VPH)

When all the required soil for a sampling location has been obtained, the soil can be homogenized as described in section 7.2. Collect sufficient volume to fill all of the remaining sample containers at least ¾ full for all other analyses. Homogenize the soil in a decontaminated stainless steel bowl, removing rocks, sticks, or other non-soil objects and breaking apart any lumps of soil prior to filling the remaining sample containers.

NOTE: Soil samples must contain greater than 30% solids for the data to be considered valid.

8.0 Quality Control and Assurance

8.1 Sampling personnel should follow specific quality assurance guidelines as outlined in the SAP. Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements outlined in the SAP typically suggest the collection of a sufficient quantity of field duplicate, field blank, and other samples.

8.2 Quality control requirements are dependent on project-specific sampling objectives. The SAP will provide requirements for equipment decontamination (frequency and materials), sample preservation and holding times, sample container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, field blanks, equipment blanks, and field duplicate samples.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

9.0 Records, Data Analysis, Calculations

All data and information (e.g., sample collection method used) must be documented on field data sheets, boring logs, or within site logbooks with permanent ink. Data recorded may include the following:

- Weather conditions;
- Arrival and departure time of persons on site;
- Instrument type, lamp (PID), make, model and serial number;
- Calibration gas used;
- Date, time and results of instrument calibration and calibration checks;
- Sampling date and time;
- Sampling location;
- Samples collected;
- Sampling depth and soil type;
- Deviations from the procedure as written; and
- Readings obtained.

10.0 Attachments or References

SOP 3-06, *Equipment Decontamination*

SOP 3-19, *Headspace Screening for Total VOCs*

SOP 3-20, *Operation and Calibration of a Photoionization Detector*

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Standard Operating Procedure SOP-3-24
Water Quality Parameter Testing for Groundwater Sampling

1.0 PURPOSE

This standard operating procedure (SOP) represents minimum standard of practice. State and federal requirements may vary, and this SOP does not replace state and federal requirements that must be consulted before work begins. Further, if a project-specific work plan has been created, the work plan should be considered the ruling document. This SOP may be modified to meet specific regulatory, client, or project specific criteria.

If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to water quality parameter testing, then those procedures may be added as an appendix to the project-specific Sampling and Analysis Plan (SAP).

2.0 SCOPE

This procedure provides guidance for expected sampling methods and protocols by all personnel related to the measurement of water quality parameters.

Field measurements of water quality parameters are commonly performed to evaluate surface water and groundwater. These tests are often performed to evaluate basic water quality parameters, to evaluate natural attenuation parameters, and to assess the presence of pore water entering a well.

As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by either the Contract Task Order (CTO) Manager or the Quality Assurance (QA) Manager, and documented.

3.0 DEFINITIONS

3.1 Barometric Pressure (BP)

The density of the atmosphere, which varies according to altitude and weather conditions.

3.2 Conductivity/Specific Conductance

A measure of the ability of water to pass electrical current, which increases with the amount of dissolved ionic substances (i.e., salts). Conductivity is inversely related to the resistance of a solution and is measured in units of mhos per centimeter (mhos/cm) (inverse ohms/cm, Siemens/cm). The conductivity of water increases with increasing temperature.

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET

Specific Conductance is corrected for 25 degrees Celsius (°C); for this reason, it is best to record Specific Conductance. If Conductivity is recorded, the temperature of the sample MUST recorded.

3.3 Dissolved Oxygen (DO)

The amount of oxygen present in water and available for respiration. DO is typically measured in milligrams per liter (mg/L). Oxygen is less soluble in warm and salty waters, so the instrument compensates the apparent percent saturation for changes in temperature and conductivity. Most probes measure the current resulting from the electrochemical reduction of oxygen (at a gold cathode) diffusing through a selective membrane. Because oxygen is being removed from the sample to perform the measurement, sample flow is required to prevent false low readings due to depletion of oxygen in the solution in front of the probe. Optical DO probes do not remove oxygen from the sample and are less affected by salts. The common range of DO in groundwater is 0.0 to 3.0 mg/L. Measurements outside of this range suggest that the meter may not be operating correctly.

3.4 Nephelometric Turbidity Unit (NTU)

The measurement of light passing through a sample based on the scattering of light caused by suspended particles.

3.5 pH

A measure of acidity and alkalinity of a solution using a logarithmic scale on which a value of 7 represents neutrality, lower numbers indicate increasing acidity, and higher numbers are increasingly basic.

3.6 Oxidation-Reduction Potential (ORP)

Also known as redox or eH, ORP is a measurement of the potential for a reaction to occur, which generally indicates the oxygen status of a sample. The probe consists of a platinum electrode, the potential of which is measured with respect to a reference electrode that rapidly equilibrates with the potential of the sample solution. A positive value indicates that oxygen is present. A negative value indicates an anaerobic environment or reducing condition. For this reason, negative ORP readings should be associated with DO readings of less than 0.5 mg/l; with negative ORP readings the water may exhibit a sulfur odor or gray color. Positive ORP readings should be associated with DO readings greater than 0.5 mg/L and lack of sulfur odors. Because of the complex relationship between ORP and temperature, no compensation is attempted; it is thus best to report both the ORP and temperature of a water sample.

3.7 Total Dissolved Solids

A measure of the quantity of materials in water that are either dissolved or too small to be filtered.

3.8 Turbidity

Measure of the clarity of water in NTUs. Potable water typically has NTU values between 0.0 and 0.3 NTUs, depending on the state or regulatory program.

4.0 RESPONSIBILITIES

The CTO Manager, or designee, is responsible for ensuring that these standard groundwater sampling activities are followed and shall review all groundwater sampling forms at the conclusion of a sampling event. The CTO Manager is responsible for ensuring that all personnel involved in monitoring well sampling shall have the appropriate education, experience, and training to perform their assigned tasks. The QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure. The Field Manager is responsible for ensuring that all project field staff follows these procedures.

Field sampling personnel are responsible for the implementation of this procedure. Personnel are required to be knowledgeable of the procedures in this SOP. Training and familiarization with this SOP shall be documented in the training file for each employee. The field sampler and/or Field Manager is responsible for directly supervising the calibration procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the CTO Manager, QA Manager, or Technical Director and then documented in the field logbook and associated report or equivalent document.

5.0 PROCEDURES

5.1 Purpose

The procedures will vary depending on parameters being measured, method of sampling, and the method of measurement used. The information here is a general guidance and the site-specific documents and manufacturer manuals supersede these procedures.

5.2 Cautions

Improper use of water quality testing equipment could result in equipment damage or compromised sampling results. Personnel should be trained to operate the test equipment being used for a field operation and should be trained in the proper techniques for collecting and

logging water quality parameters. Personnel should also be able to recognize problems with test equipment and have someone available for basic troubleshooting and repair.

5.3 Interferences

During field testing, water quality data that is documented from field testing equipment may be influenced by certain outside factors that are unrelated to the actual site water quality. Such parameters and equipment include the following:

pH Meters

- Coatings of oils, greases, and particles may impair the electrode's response. Pat the electrode bulb dry with lint-free paper or cloth and rinse with de-ionized water. For cleaning hard-to-remove films, use isopropyl alcohol very sparingly so that the electronic surface is not damaged.

- Poorly buffered solutions with low specific conductance (less than 200 microsiemens per centimeter) may cause fluctuations in the pH readings. Equilibrate electrode by immersing in several aliquots of sample before taking pH.

Dissolved Oxygen

- Dissolved gases (e.g., hydrogen sulfide, halogens, sulfur dioxide) are a factor with the performance of DO probes. The effect is less pronounced on optical DO meters. Meter type and potential interferences should be considered based on potential sulfate/sulfide or nitrate/nitrite reducing environments.

- Exposure of the sample to the atmosphere will cause elevated DO measurements.

Turbidity Meter

- If the weather is warm and humidity is high, condensation may collect on the cuvet. To avoid this, allow the sample to warm and dry the outside of the cuvet before making the measurement. One method used to accomplish this is to place the cuvet against one's body (armpits work well).

Temperature

- Sample temperature will change rapidly when there are significant differences between the sample and ambient air.

5.4 Apparatus and Materials

Field personnel shall consult the site work plan and SAP to review the equipment requirements for the sampling procedures to be followed during the sampling effort. The specific apparatus and materials required will depend on the water quality parameters being monitored. Table 1 shows the common equipment used in water quality parameter testing.

Table 1
Water Quality Parameter Testing — Common Equipment

Water Quality Parameter Instrument	Calibration Standards Required	Other Equipment
pH Meter	Yes - 2 or 3 Point Standards depending on groundwater range. Calibration must cover the range to be measured. If samples are above or below typical buffer standards (4, 7 and 10), special order buffers that fall outside groundwater pH range.	Container or flow thru cell for holding sample
Specific Conductance	Yes	Container or flow thru cell for holding sample
ORP Meter	Yes	Container or flow thru cell for holding sample
Turbidity Meter	Yes	Container or flow thru cell for holding sample
DO	No	Container or flow thru cell for holding sample
Thermometer	No	Container or flow thru cell for holding sample
Flow Rate	No	Calibrated Container

Notes:
ORP = Oxidation-Reduction Potential
DO = Dissolved Oxygen

5.5 Instrument or Method Calibration

Most monitoring instruments require calibration before use, and this calibration must be conducted in the field under the ambient climatic conditions that will be present during field sampling. Calibration of monitoring instruments shall be performed in accordance with the manufacturer's specifications and recorded in the provided form in Attachment 1. Site-specific instrument calibration requirements should be specified in the SAP. The following minimum calibration requirements apply to the various types of meters used to gather water quality measurements.

Initial Calibration (IC): Before use, the instrument or meter electronics are adjusted (manually or automatically) to a theoretical value (e.g., DO saturation) or a known value of a

calibration standard. An IC is performed in preparation for the first use of an instrument or if a calibration verification does not meet acceptance criteria.

Initial Calibration Verification (ICV): The instrument or meter calibration is checked or verified directly following IC by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria for the instrument/parameter. If an ICV fails to meet acceptance criteria, immediately recalibrate the instrument using the applicable initial calibration procedure or remove it from service.

Continuing Calibration Verification (CCV): After use, the instrument or meter calibration is checked or verified by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria for the instrument/parameter.

5.5.1 Calibration Checks

Calibration checks are conducted by measuring a known standard. They must be completed after calibration and should be performed at least one other time (i.e., after lunch) and anytime suspect measurements are encountered. Table 2 provides general acceptance ranges to be used during calibration checks. If a meter is found to be outside of the acceptance range, the meter **must** be recalibrated. If the meter remains out of range, the project manager and/or the supplier of the meter should be contacted to determine alternative measures.

Table 2
Calibration Check Acceptance Limits

Parameter	Acceptance Criteria
Dissolved Oxygen	±0.3 mg/L of the theoretical oxygen solubility
Oxidation-Reduction Potential	±10 mv from the theoretical standard value at that temperature
pH	±0.2 Standard pH Units
Specific Conductance	±5% of the standard
Turbidity	0.1 to 10 NTU: ±10% of the standard 11 to 40 NTU: ±8% of the standard 41 to 100 NTU: ±6.5% of the standard

Notes:
mg/L = milligrams per liter
mv = millivolts
NTU = nephelometric turbidity units

5.5.2 Possible and Suspected Ranges

The concentration for each parameter range should be known so that concentrations outside of the range can be noted. Table 3 presents the maximum range of the parameter in groundwater. The table also presents the suspected range. Measurements outside of the maximum/minimum range should be considered in error and the measurement method should be checked. Concentrations outside the normal range should be treated as suspect but may be the result of contaminant impact. For example, a pH of 2.0 would be out of the normally suspected range for groundwater but not at a site impacted with an acid.

Table 3
Minimum and Maximum Result Ranges

Parameter	Units	Possible Min	Possible Max	Normal Min	Normal Max	Notes
Dissolved Oxygen	mg/L	0.0	14.6 (0°C) 10.1 (15°C) 8.3 (2°C)	0.0	5	The colder the sample, the higher the DO reading. DO greater than 1 mg/L, ORP positive should not have sulfur odor, sulfide, ferrous iron and/or gray color. DO less than 1 mg/L, ORP negative, may have sulfur odor, sulfide, ferrous iron and/or gray color.
pH	SU	0	14	5	9	pH values exceeding 10 could indicate grout contamination
ORP	mv					DO greater than 1 mg/L, ORP positive should not have sulfur odor, sulfide, ferrous iron and/or gray color. DO less than 1 mg/L, ORP negative, may have sulfur odor, sulfide, ferrous iron and/or gray color.
Specific Conductance	µS/cm			varies	varies	
Temperature	°C	0	100	5	30	
Turbidity	NTU	0	Greater than 1,000	0	Greater than 1,000	50 NTU or greater suggests cloudiness.

Notes:

mg/L = milligrams per liter
°C = degrees Celsius
DO = dissolved oxygen
SU = standard units
ORP = oxidation reduction potential
mv = millivolts
mS/cm = micro Siemens per cm
NTU = nephelometric turbidity units

5.5.3 Field Instruments and Calibration Criteria

The calibration acceptance criteria for each instrument are summarized in Table 4 along with special considerations related to each field instrument.

Table 4
Calibration Check Acceptance Limits

Parameter	Acceptance Criteria
Dissolved Oxygen	±0.3 mg/L of the theoretical oxygen solubility.
Oxidation-Reduction Potential	±10 mv from the theoretical standard value at that temperature.
pH	±0.2 Standard pH Units
Specific Conductance	±5% of the standard
Turbidity	0.1 to 10 NTU: ±10% of the standard 11 to 40 NTU: ±8% of the standard 41 to 100 NTU: ±6.5% of the standard

Notes:
mg/L = milligrams per liter
mv = millivolts
NTU = nephelometric turbidity units

pH Meters

- For the most accurate of pH measurements, pH meters should receive a three-point calibration. However, if a two-point calibration will bracket the groundwater pH of the site, a two-point calibration is acceptable. Three-point calibrations typically include calibrating to solutions of pH 7.00, 4.00, and 10.00. If groundwater pH is outside the calibration range of the solution standards, special buffers must be ordered to bracket the pH. Some meters will report the slope of the calibration and this may be used in checking the meter calibration (refer to the meter's manual). When performing an ICV, the result must be within +/- 0.2 pH units of the stated buffer value.

- pH meters should be calibrated across the range of values to be measured. The maximum and minimum calibration solutions shall be outside the range of anticipated values. For example, if the expected range is between 7.50 and 9.00, the 7.00 and the 10.00 standard should be used for calibration. Perform the IC using at least two buffers, and always use the pH 7.00 buffer first. A reading that is above the maximum (or below the minimum) calibration standard is an estimate only and is not valid. This condition requires obtaining a new standard that is above (or below) the reported value, depending on the measurement.

- A percent slope of less than 90 percent indicates a bad electrode that must be changed or repaired. If percent slope cannot be determined, or the manufacturer's optimum specifications are different, follow the manufacturer's recommendation for maintaining optimum meter performance.

Specific Conductivity Meters

- For IC, when the sample measurements are expected to be 100 microsiemens per centimeter (µS/cm) or greater, use two standard potassium chloride (KCl) solutions that bracket the range of expected sample conductivities. Calibrate the instrument with the first standard. Verify the calibration of the instrument with the second standard, bracketing the range of expected sample values.

- If the instrument can be calibrated with more than one standard, choose additional calibration standards within the range of expected sample values.

- When the sample measurements are expected to be less than 100 µS/cm, a lower bracket is not required, but one standard (KCl) solution that is within the range of expected measurements must be used for the IC and the ICV.

- Accept the calibration if the meter reads within +/- 5 percent of the value of any calibration standard used to verify the calibration.

- Most field instruments read conductivity directly. Record all readings and calculations in the calibration records.

- For CCV, check the meter with at least one KCl standard with a specific conductance in the range of conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5 percent of the standard value.

- If new environmental samples are encountered outside the range of the IC, verify the instrument calibration with two standards bracketing the range of sample values. If these calibration verifications fail, recalibrate the instrument.

Dissolved Oxygen Meters

- Before calibrating, check the probe membrane for bubbles, tears, or wrinkles. These conditions require replacement of the membrane in accordance with the manufacturer's directions.

- If the meter provides readings that are off-scale, will not calibrate, or drift, check the leads, contacts, etc., for corrosion and/or short circuits. These conditions require replacement maintenance in accordance with the manufacturer's directions.
- Most DO meters must be calibrated based on an environment of 100 percent humidity and a known elevation and barometric pressure (BP).

- For 100 percent humidity, place the probe in the calibration container with a moist towel and allow the probe to remain, undisturbed, for 10 to 20 minutes.

- The IC is an air calibration at 100% saturation. Before use, verify the meter calibration in water-saturated air to make sure it is properly calibrated and operating correctly. Make a similar verification at the end of the day or sampling event. Follow the manufacturer's instructions for your specific instrument. Allow an appropriate warm up period before IC. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops), wipe any droplets off the membrane/sensor and insert the sensor into the chamber (this ensures 100 percent humidity). Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate. Once the probe/calibration chamber is stable at ambient temperature, check the air temperature and determine, from the DO versus temperature table (see Attachment 2) what DO should measure. The acceptance criterion for DO ICV is +/- 0.3 mg/L.

- Use the same procedure as above for CCV.

ORP Meters

- Verify electrode response before use in the field.

- Equilibrate the standard solution to the temperature of the sample. The standard solution is based on a 25°C temperature; however, the calibration solution standard's value will require adjustment based on the temperature.

- Immerse the electrodes and gently stir the standard solution in a beaker (or flow cell). Turn the meter on, placing the function switch in the millivolt (mv) mode.

- Let the electrode equilibrate and record the reading to the nearest millivolt. The reading must be within ±10 mv from the theoretical redox standard value at that temperature. If not, determine the problem and correct it before proceeding. Switch to temperature display and read the value.

- Record the mv reading and temperature in the field notebook or in form. Rinse the electrode with distilled water and proceed with the sample measurement, unless using a flow cell. If a flow cell is used, rinse between sample locations.

Turbidity Meters

- Perform an initial calibration using at least two primary standards.

- If the instrument cannot be calibrated with two standards, calibrate the instrument with one standard and verify with a second standard.

- Perform an ICV by reading at least one primary standard as a sample. The acceptance criterion for the ICV depends on the range of turbidity of the standard value:

    1. Standard Value = 0.1 to 10 NTU: the response must be within 10 percent of the standard;

    2. Standard Value = 11 to 40 NTU: the response must be within 8 percent of the standard;

    3. Standard Value = 41 to 100 NTU: the response must be within 6.5 percent of the standard; and

    4. Standard Value greater than 100 NTU: the response must be within 5 percent of the standard.

- Determining the Values of Secondary Standards: Use only those certified by the manufacturer for a specific instrument. Secondary standards may be used for CCVs.

To initially determine the value of a secondary standard, assign the value that is determined immediately after an ICV or verification with primary standards. This is done by reading the secondary standard as a sample. This result must be within the manufacturer's stated tolerance range and +/- 10 percent of the assigned standard value. If the +/- 10 percent criterion is not met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

- CCV: Perform a CCV using at least one primary or secondary standard. The calibration acceptance criteria are the same as those for an ICV.

5.6 Direct Measurements

Direct measurements with meters are the most common methods and can be accomplished by placing a sample in a container with the probe or by allowing the water to flow past the probe in a flow cell. The use of a flow-through cell improves measurement quality by allowing the constant flow of water over the probes and reduces interaction of the sample with the atmosphere. Sample cups should be avoided. The quantity of samples, timing, and methodology should be described in the project SAP.

Following calibration of required probes, connect the bottom flow-cell port to the discharge line of the pump. Connect the top port to a discharge line directed to a bucket to collect the purge water. Allow the flow cell to completely fill. As the water flows over the probe, record the measurements. Continue to record the measurements at regular intervals, as specified in the SAP.

When the ambient air temperatures are much higher or lower than the temperature of the water sample, it is best to keep the length of tubing between the wellhead and the flow cell as short as possible to prevent heating or cooling of the water. Tubing and flow-through cell should not be exposed to direct sunlight, particularly in the summer, if at all possible, to avoid heating of water samples.

5.7 Data Acquisitions, Calculations, and Data Reduction

5.7.1 Specific Conductivity Correction Factions

If the meter does not automatically correct for temperature (i.e., read Specific Conductivity) record Conductivity and adjust for temperature upon returning to the office. The following equation can be used to convert Conductivity to Specific Conductivity.

$$K = \frac{(Km)(C))}{1 + 0.0191(\mathrm{T} - 25)}$$

Where:

K = Conductivity in μmhos/cm at 25°C
Km = Measured conductivity in μmhos/cm at T degrees Celsius
C = Cell constant
T = Measured temperature of the sample in degrees Celsius;

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(Km)}{1 + 0.0191(\mathrm{T} - 25)}$$

5.7.2 Percentage Difference Calculation

For evaluating slope of readings from either a flow cell or a sample cup.

$$\%Difference = \frac{(Highest\ Value - Lowest\ Value)}{(Highest\ Value)}\ x\ 100$$

5.7.3 Convert mm mercury (mmHG) to inches mercury (inHG)

$$mmHG = inHG\ x\ 25.4$$

5.7.4 True Barometric Pressure

For converting BP obtained from a public domain source that is expressed in BP at sea level to BP at the subject site.

$$TrueBP = (BP) - \frac{(2.5\ x\ [Local\ Altitude])}{100}$$

Where: BP is in mmHG and Local Altitude is in feet

Example: BP at site A is 30.49 inHg and elevation is 544 feet, calculate TrueBP

Convert inHG to mmgHG:

mmHg = 30.49 inHg x 25.4 = 774.4 mmHg

Calculate True BP:

TrueBP = (774.4 mmHg) – [2.5 * (544 /100)] = 774.4-13.6 = 760.8 mmHg

6.0 RECORDS

Data will be recorded promptly, legibly, and in indelible ink on the appropriate logbooks and forms. At the completion of a field effort, all logbooks, field data forms, and calibration logs shall be scanned and made electronically available to the project team. The original field forms, calibrations logs, and log book will be maintained in the project file.

7.0 HEALTH AND SAFETY

Detailed Health and Safety requirements can be found in the site specific Health and Safety Plan. Ensure that a Safe Work Assessment and Permit form is filled out daily prior to any work in the field and reviewed with all project personnel in a daily safety brief.

Safety glasses with side shields or goggles and disposable gloves shall be worn during calibration activities.

8.0 REFERENCES

None

9.0 ATTACHMENTS

Attachment 1: Example Field Instrument Calibration Form
Attachment 2: Solubility of Oxygen at Given Temperatures
Attachment 3: Example Field Data Form

Attachment 1
Example Field Instrument Calibration Form

Field Instrument Calibration Form

Calibrated by: _____ Equipment (Make/Model/Serial#): _____

Date: _____ Equipment (Make/Model/Serial#): _____

pH (su) — Standard: ± 0.2 standard units

Initial Calibration

	Hach SL	Reading	Pine SL	Reading
pH7				
pH4				

Continuing Calibration Verification

	Hach SL	Reading	Deviation	Acceptable Variance (Y/N)
pH7				
pH4				

DO (mg/L) — Standard: ± 0.3 mg/L of theoretical*

IC (Temp:) **ICV (Temp:)**

Saturation (%)	Reading (%)	Theoretical (mg/L)	Reading (mg/L)
100			

CCV (Temp:)

Saturation (%)	Reading (%)	Deviation	Acceptable Variance (Y/N)
100			

Theoretical (mg/L)	Reading (mg/L)	Deviation	Acceptable Variance (Y/N)

ORP (mV) — Standard: NA

IC (Zobell SL:) **ICV (Pine SL:)**

TCS (Std/Temp)	Reading	TCS (Std/Temp)	Reading

CCV (Zobell SL:)

TCS (Std/Temp)	Reading	Deviation	Acceptable Variance (Y/N)

Turbidity (ntu) — Standard: ±10% of Standard

Initial Calibration

Standard	Reading

Continuing Calibration Verification

Standard	Reading	Deviation	Acceptable Variance (Y/N)

Conductivity (ms^{C}/cm) — Standard: ± 5% of standard value

IC (YSI SL:) **ICV (Pine SL:)**

Standard	Reading	Standard	Reading

CCV (YSI SL:)

Standard	Reading	Deviation	Acceptable Variance (Y/N)

Comments:

Notes:

SL	solution lot		su	standard units		ntu	Nephelometric Turbidity Units
TCS	temperature corrected standard		mV	millivolts		°C	degrees Celsius
Std	standard		%	percent		ms^{C}/cm	millisiemens per centimeter (temperature corrected)
Temp	temperature		mg/L	milligrams per liter		*	Theoretical value

Attachment 2
Solubility of Oxygen at Given Temperatures

Field Measurement of Dissolved Oxygen

Solubility of Oxygen in Water at Atmospheric Pressure			
Temperature	**Oxygen Solubility**	**Temperature**	**Oxygen Solubility**
°C	**mg/L**	**°C**	**mg/L**
0.0	14.621	26.0	8.113
1.0	14.216	27.0	7.968
2.0	13.829	28.0	7.827
3.0	13.460	29.0	7.691
4.0	13.107	30.0	7.559
5.0	12.770	31.0	7.430
6.0	12.447	32.0	7.305
7.0	12.139	33.0	7.183
8.0	11.843	34.0	7.065
9.0	11.559	35.0	6.950
10.0	11.288	36.0	6.837
11.0	11.027	37.0	6.727
12.0	10.777	38.0	6.620
13.0	10.537	39.0	6.515
14.0	10.306	40.0	6.412
15.0	10.084	41.0	6.312
16.0	9.870	42.0	6.213
17.0	9.665	43.0	6.116
18.0	9.467	44.0	6.021
19.0	9.276	45.0	5.927
20.0	9.092	46.0	5.835
21.0	8.915	47.0	5.744
22.0	8.743	48.0	5.654
23.0	8.578	49.0	5.565
24.0	8.418	50.0	5.477
25.0	8.263		

Notes:

The table provides three decimals to aid interpolation

Under equilibrium conditions, the partial pressure of oxygen in air-saturated water is equal to that of the oxygen in water saturated

°C = degrees Celsius

mg/L = milligrams per liter

Attachment 3
Example Field Data Form

WELL DEVELOPMENT & GROUNDWATER SAMPLING FORM

DATE:	JOB NUMBER:	EQUIPMENT (Make/Model #/Serial #):
PROJECT:	EVENT:	/ /
WELL ID:	LOCATION:	/ /
WEATHER CONDITIONS:	AMBIENT TEMP:	/ /
REVIEWED BY:	PERSONNEL:	/ /

WELL DIA:	**WELL DEVELOPMENT**	
TOTAL DEPTH from TOC (ft.):	START:	FINISH:
DEPTH TO WATER from TOC (ft.):	VOLUME PURGED (gal):	
LENGTH OF WATER COL. (ft.):	**GROUNDWATER SAMPLING**	
1 VOLUME OF WATER (gal):	START:	FINISH:
3 VOLUMES OF WATER (gal):	VOLUME PURGED (gal):	
	ANALYSIS:	

WELL DEVELOPMENT PARAMETERS		GW SAMPLING PARAMETERS	
Temperature:	± 1.0° C	Temperature:	± 0.2° C
pH:	± 0.5 standard units	pH:	± 0.2 standard units
Specific Conductance:	± 10% of the past measurement	Specific Conductance:	± 5% of the past measurement
Turbidity:	relatively stable	DO:	≤ 20% saturation
		ORP:	± 10 millivolts
		Turbidity:	≤ 10 NTU

IN-SITU TESTING

Circle one: DEVELOPMENT SAMPLING							☐ Bailer ☐ Pump		Description:			
Time (hh:mm):												
pH (units):												
Conductivity (mS/cm):												
Turbidity (NTU):												
DO (mg/L): YSI 556												
DO (mg/L): YSI 550												
Temperature (C°):												
ORP (mV):												
Volume Purged (gal):												
Depth to Water (ft):												
									Well Goes Dry While Purging ☐			

SAMPLE DATA ☐ Bailer ☐ Pump Description:

Sample ID	Date (m/d/y)	Time (hh:mm)	Bottles (total to lab)	Filtered (0.45 µm)	Remarks
Purging/Sampling Device Decon Process:					

COMMENTS:

Purge water placed in drum#_____

Subsurface Soil Sampling by Split Spoon

Procedure 3-33

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the methods used in obtaining subsurface soil samples using a split spoon for physical and/or chemical analysis. Subsurface soil samples are obtained in conjunction with soil boring programs and provide information as to the physical and/or chemical makeup of the subsurface environment. Specific information regarding sampling locations can be found in the associated Sampling and Analysis Plan (SAP).

1.2 The purpose of this SOP is to provide a description of a specific method or procedure to be used in the collection of subsurface soil samples. Subsurface soil is defined as unconsolidated material which may consist of one or a mixture of the following materials: sand, gravel, silt, clay, peat (or other organic soils), and fill material. Subsurface soil sampling, conducted in accordance with this SOP will promote consistency in sampling and provide a basis for sample representativeness.

1.3 This SOP covers subsurface soil sampling by split-spoon only, as this is the means most often used for obtained samples of unconsolidated deposits. Other types of equipment are available for use in subsurface soil sampling, including thin-wall tube samplers (Shelby tubes), piston samplers, and continuous core barrel samplers. Information on the use of these other sampling devices may be found in several available drilling handbooks and respective state and/or federal agency technical guidance documents. The American Society for Testing and Materials (ASTM) also provides procedures for use of split-spoon and other sampling devices.

1.4 Split-spoon subsurface soil sampling generally requires use of a drilling rig and typically the hollow-stem auger (HSA) or other common drilling method to generate a borehole in which to use the split-spoon sampler. The split-spoon sampler is inserted through the augers (or other type of drill casing) then is driven into the subsurface soil with a weighted hammer. The sampler is then retrieved and opened to reveal the recovered soil sample. Soil samples may be collected at a continuous interval or at pre-selected vertically spaced intervals within the borehole.

1.5 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).

1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific SAP.

1.7 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first location. All **field sampling personnel** responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety Officer (SSO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the HASP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSO.

2.4 The health and safety considerations for the work associated with split spoon sampling include:

- To avoid lifting injuries associated with carrying split spoons and associated drilling/sampling equipment, use large muscles of the legs, not the back.
- Stay clear of all moving equipment, and avoid wearing loose fitting clothing.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and in case of extreme cold, wear fitted insulated clothing.
- Be aware of restricted mobility due to PPE.
- At no point should soil samples be put into the mouth.

3.0 Terms and Definitions

None.

4.0 Interference

Potential interferences could result from cross-contamination between samples or sample locations. Minimization of the cross contamination will occur through the following:

- The use of clean sampling tools at each location as necessary.
- Avoidance of material that is not representative of the media to be sampled.
- Extreme temperatures or humidity can affect the reliability of certain field screening equipment, such as photoionization detectors (PID).
- If screening soil samples for headspace readings in extreme cold temperatures, samples may need to be warmed slightly prior to screening.

5.0 Training and Qualifications

5.1 Qualifications and Training

5.1.1 The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **Contract Task Order (CTO) Manager** is responsible for ensuring that split spoon sampling activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all field sampling personnel involved in split spoon sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager** (FM) is responsible for ensuring that all field sampling personnel follow these procedures.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure. Minimum qualifications for field sampling personnel require that one individual on the field team shall have a minimum of 6 months of experience with split spoon sampling.

5.2.5 The **field sampler and/or task manager** is responsible for conducting split spoon soil sampling in a manner which is consistent with this SOP. The field sampler or designee will observe all activities pertaining to split spoon soil sampling to ensure that the SOP is followed, and to record all pertinent data onto a boring log. It is also the responsibility of the field sampler or designee to indicate the specific targeted sampling depth or sampling interval to the drilling subcontractor. The field sampler or designee is also responsible for the collection of representative environmental or stratigraphic characterization samples once the sampling device has been retrieved and opened. Additional sample collection responsibilities include labelling, handling, and storage of samples until further chain-of-custody procedures are implemented**.** If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the Program Quality Manager and then documented in the field logbook and associated report or equivalent document.

5.2.6 It will be the responsibility of the **drilling subcontractor** to provide the necessary materials for obtaining subsurface soil samples. This generally includes one or more split-spoon samplers in good operating condition and sample containers used for stratigraphic characterization samples (sample containers for environmental samples should be provided by the designated analytical laboratory). It is the drilling subcontractor's responsibility to provide and maintain their own boring logs if desired. Equipment decontamination materials should also be supplied by the subcontractor and should meet project specifications.

5.2.7 Sampling personnel will also be health and safety trained and certified as specified in the HASP.

6.0 Equipment and Supplies

In addition to those materials provided by the subcontractor, the following equipment list contains materials which may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- SAP and HASP
- Personal protective equipment (PPE) and other safety equipment, as required by the HASP
- Boring logs
- Teaspoon or spatula (stainless steel is recommended)

- Sample kit (bottles, labels, custody records and tape, cooler with ice)
- Folding rule or tape measure
- Equipment decontamination materials
- Geologist Table
- Field logbook and pen with indelible ink

7.0 Calibration or Standardization

Field screening instruments (eg. PIDs) should be calibrated following manufacturer's instructions prior to the start of work and as a set time interval throughout the day. Calibration information should be recorded in the field log book or on a calibration log form.

8.0 Procedure

8.1 General Method Description

Split-spoon sampling devices are typically constructed of steel and are most commonly available in lengths of 18 and 24 inches and diameters of 1.5 to 3 inches. The split-spoon consists of a tubular body with two halves that split apart lengthwise, a drive head on the upper end with a ball-check valve for venting, and a hardened steel cutting shoe at the bottom. The soil sample enters the split-spoon through the cutting shoe as the device is driven into the ground. A replaceable plastic or metal basket is often inserted into the shoe to assist with retaining samples. Once the sampler is retrieved, the drive head and cutting shoes are removed and the split-spoon halves are then separated, revealing the sample.

Sample depth intervals are usually defined on a project-specific basis with these requirements specified in the project sampling plan. Sampling intervals typically range from one (1) sample per five (5) feet of drilling to continuous sampling where the entire drilled interval is sampled.

Subsurface soil sampling is usually accomplished as part of a drilling program where a soil boring is advanced with drilling equipment to the designated depth prior to collection of a representative sample. The general procedures outlined briefly in the following section provide requirements for advancing drill casing/augers in preparation for sampling.

8.2 General Procedures - Borehole Preparation

8.2.1 Advancing Casing/Augers

Soil borings that are completed for soil sampling purposes are typically advanced using hollow-stem augers and sometimes drive-and-wash or other casing methods. The casing/augers must be of sufficient diameter to allow for soil sampling at a minimum. The casing/augers will be advanced according to project requirements to the required depth for sampling. If hollow-stem augers are used, a temporary plug shall be used in the lead auger to prevent the auger from becoming filled with drill cuttings while drilling is in progress.

8.2.2 Obstructions

For those borings which encounter obstructions, the casing/augers will be advanced past or through the obstruction if possible. Caution should be exercised when obstructions are encountered and an effort made to identify the obstruction before drilling is continued. If the obstruction is not easily drilled through or removed, the boring should be relocated to an adjacent location.

8.2.3 Use of Added Water

The use of added or recirculated water during drilling is permitted when necessary. Use of extraneous water should be minimized or avoided if possible as it may impact sample quality. Water usage should be documented in the field notebook. Sampling and analysis of added or recirculated water may be

required for quality assurance purposes (refer to SAP). If a well is installed within the completed borehole, removal of the added water may be required. State and/or local permitting may be required for adding water to the subsurface.

8.3 Sampling Procedure

8.3.1 Equipment Decontamination

Each split-spoon must be decontaminated prior to its initial use and following collection of each soil sample. Equipment decontamination procedures should be performed in accordance with SOP 3-06 – Equipment Decontamination. Any additional site-specific requirements for equipment decontamination will be outlined in the SAP.

8.3.2 Standard Penetration Test

The drilling subcontractor will lower the split-spoon into the borehole. Samples are generally obtained using the Standard Penetration Test (SPT) in accordance with ASTM standards (ASTM D 1586-84). Following this method, the sampler will be driven using the 140-pound hammer with a vertical free drop of 30 inches using two turns of the rope on the cathead. The number of hammer blows required for every 6 inches of penetration will be recorded on the boring log. Blowcount information is used as an indicator of soil density for geotechnical as well as stratigraphic logging purposes. Once the split-spoon has been driven to its fullest extent, or to refusal, it will be removed from the borehole.

8.3.3 Sample Recovery

The split-spoon will be immediately opened upon removal from the casing/auger. The open sampler should be screened for volatile organics with a photoionization device (PID) if required by the SAP. If the SAP also requires individual soil sample headspace screening for volatile organic compounds (VOCs), the procedure should be performed according to SOP 3-19 Headspace Screening for VOCs.

Sample recovery will be determined by the project geologist or designee who will examine the soil core once the sampler is opened. The length of sample shall then be measured with a folding rule or tape measure. Any portion of the split-spoon contents which are not considered part of the true sample (i.e., heaved soils) will be discarded. If the sample recovery is considered inadequate for sample characterization or analytical testing purposes, another sample should be collected from the next vertical interval if possible before drilling is reinitiated.

Adequate sample recovery for stratigraphic logging and/or headspace screening for VOCs purposes should be approximately 6 inches. Adequate sample recovery for analytical testing purposes should be a minimum of 12 inches and is somewhat dependent on the type of analytical testing required. In some cases, continuous sampling over a short interval, and compositing of the sample, may be required to satisfy analytical testing requirements. Larger diameter samplers may be used if large volumes of soil are required for analytical testing.

8.3.4 Sample Containment - General

Once retrieved, the sample will be removed from the split-spoon with a teaspoon or spatula and placed into the appropriate sample container. The sample will be split if necessary to meet sampling program requirements. Sample splitting may be necessary to provide individual samples for headspace testing, visual characterization, physical testing, analytical testing, or simply for archiving purposes. In general, most sampling programs are structured around environmental characterization needs; therefore, sample portions required for analytical testing should be collected first. The SAP will provide specific sample container requirements for each type of sample and should be referred to for guidance.

Once filled, the sample containers should be properly capped, cleaned, and labeled, and chain-of-custody and sample preservation procedures initiated. Sampling equipment should then be properly decontaminated.

8.3.5 Sample Containment - Volatile Organic Analyses

Collection of subsurface soil samples for VOCs is more complex than collection of samples for other routine chemical or physical testing primarily because of the concern for the potential loss of volatiles during the sample collection procedure. To limit the potential for loss of volatiles, the soil sample needs to be obtained as quickly and as directly as possible from the split-spoon. This generally means that the VOC sample is collected and placed into the appropriate sample container first. The VOC sample should also be obtained from a discrete portion of the entire sample interval and not composited or homogenized. The remainder of the recovered sample can then be composited, homogenized or split to meet the other testing requirements. The boring log and/or sample logbook should be filled out to indicate actual sample collection depths for both VOA samples and other portions of the sample which may have been composited over a larger vertical interval.

9.0 Quality Control and Assurance

9.1 Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific SAP. Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements outlined in the SAP typically suggest the collection of a sufficient quantity of field duplicate, field blank, and other samples.

9.2 Quality control requirements are dependent on project-specific sampling objectives. The SAP will provide requirements for sample preservation and holding times, sample container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, field blanks, equipment blanks, and field duplicate samples.

10.0 Data Records and Management

10.1 Records will be maintained in accordance with SOP 3-03 Recordkeeping, Sample Labelling, and Chain-of-Custody. Various forms are required to ensure that adequate documentation is made of sample collection activities. These forms include:

- Boring logs and/or electronic data collection devices
- Field log books
- Chain-of-custody records
- Shipping labels

Original copies of these records should be maintained in the appropriate project files.

10.2 Boring logs (Attachment 1) will provide visual and descriptive information for each sample collected and are often the most critical form of documentation generated during a sampling program. Field electronic data collection devices (e.g., Yuma Tablet, Toughbook Laptop) with data collection software may also be used to record boring log information.

10.3 The field log book is kept as a general log of activities.

10.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.

10.5 Shipping labels are required if sample coolers are to be transported to the laboratory by a third party (courier service).

11.0 Attachments or References

Attachment 1 – Boring Log

ASTM D 1586-84 Subsurface Exploration Using the Standard Penetration Test and the Cone Penetrometer Test

SOP 3-03 Recordkeeping, Sample Labeling, and Chain-of-Custody

SOP 3-06 Equipment Decontamination

Author	*Reviewer*	*Revisions (Technical or Editorial)*
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Attachment 1
Boring Log

RESOLUTION CONSULTANTS

Client:		Boring ID:
Project Number:		
Site Location:		
Coordinates:	Elevation:	Sheet: 1 of 1
Drilling Method:		Monitoring Well Installed:
Sample Type(s):	Boring Diameter:	Screened Interval:
Weather:	Logged By: / Date/Time Started:	Depth of Boring:
Drilling Contractor:	Ground Elevation: / Date/Time Finished:	Water Level:

Depth (ft)	Geologic sample ID	Sample Depth (ft)	Blows per 6"	Recovery (inches)	Headspace (ppm)	U.S.C.S	MATERIALS: Color, size, range, MAIN COMPONENT, minor component(s), moisture content, structure, angularity, maximum grain size, odor, and Geologic Unit (If Known)	Lab Sample ID	Lab Sample Depth (Ft.)
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									

NOTES:

Date	Time	Depth to groundwater while drilling

Checked by _____ Date:

PRINTED COPIES ARE UNCONTROLLED. CONTROLLED COPY IS AVAILABLE ON COMPANY INTRANET.

Appendix C

Laboratory Certifications

State of Rhode Island Certification

State of Rhode Island and Providence Plantations
DEPARTMENT OF HEALTH
Certifies

LAO00303

KATAHDIN ANALYTICAL SERVICES INC
600 TECHNOLOGY WAY
SCARBOROUGH ME 04074
Laboratory Director: DEBORAH NADEAU

for the analysis of: **Potable Water Organic Chemistry - Potable Water Inorganic Chemistry - Non-potable Water Organic Chemistry - Non-potable Water Inorganic Chemistry -**

This certificate is issued, pursuant to Rhode Island General Laws 23-16.2 and supersedes all previous Rhode Island certificates issued to this laboratory. Certification is no guarantee of the validity of the laboratory results.

This certificate is valid only when accompanied by the certificate and list of analytes and methods for which certification has been granted based upon the following out of state certification(s):

Certifying Authority	*Certification Number*	*Expiration Date*
MAINE	*ME0019*	*06/01/2014*
NH	*200112*	*04/02/2013*

KATAHDIN ANALYTICAL SERVICES INC is responsible for maintaining each of the certifications listed above. Failure to notify the Laboratory Certification Officer of any change in the status of these certifications may result in the suspension or revocation of certification. Contact the Laboratory Certification Officer to verify the current certification status of this laboratory.

Michael Fine, MD
Director of Health

Expires: 12/30/2013

THIS CERTIFICATE IS TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY IN A LOCATION VISABLE TO THE PUBLIC

Katahdin Analytical Certifications

DoD ELAP Certification

LABORATORY ACCREDITATION BUREAU®

Certificate of Accreditation

ISO/IEC 17025:2005 Certificate Number L2223

Katahdin Analytical Services, Inc.

600 Technology Way
Scarborough ME 04074

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation valid through: February 1, 2016

R. Douglas Leonard, Jr., President, COO
Laboratory Accreditation Bureau
Presented the 1st of February 2013

*See the laboratory's Scope of Accreditation for details of accredited parameters
**Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Scope of Accreditation
For
Katahdin Analytical Services, Inc.

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
207-874-2400

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.2) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Katahdin Analytical Services to perform the following tests:

Accreditation granted through: February 1, 2016

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	2, 4`-DDD
GC/ECD	EPA 8081B	2, 4`-DDE
GC/ECD	EPA 8081B	2, 4`-DDT
GC/ECD	EPA 608; EPA 8081B	4, 4`-DDD
GC/ECD	EPA 608; EPA 8081B	4, 4`-DDE
GC/ECD	EPA 608; EPA 8081B	4, 4`-DDT
GC/ECD	EPA 608; EPA 8081B	Aldrin
GC/ECD	EPA 608; EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane
GC/ECD	EPA 608; EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Cis-Nonaclor
GC/ECD	EPA 608; EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 608; EPA 8081B	delta-BHC
GC/ECD	EPA 608; EPA 8081B	Dieldrin
GC/ECD	EPA 608; EPA 8081B	Endosulfan I
GC/ECD	EPA 608; EPA 8081B	Endosulfan II

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 608; EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 608; EPA 8081B	Endrin
GC/ECD	EPA 608; EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 608; EPA 8081B	Heptachlor
GC/ECD	EPA 608; EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Hexachlorobenzene
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Oxychlordane
GC/ECD	EPA 608; EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8081B	trans-Nonachlor
GC/ECD	EPA 608; EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4, 5-Tetrachlorobiphenyl (BZ 48)
GC/ECD	EPA 8082A	2, 2', 4, 5'-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5'-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3, 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015C/D MOD	Diesel range organics (DRO)
GC/FID	EPA 8015C/D MOD	Total Petroleum Hydrocarbon (TPH)
GC/FID	EPA 8015C/D MOD	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	CT ETPH	Total Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics
GC/ECD	EPA 8011; EPA 504	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011; EPA 504	1, 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene
GC/MS	EPA 8260B/C; EPA 524.2	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1, 1-Trichloroethane
GC/MS	EPA 624; 8260B/C; EPA 524.2	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1, 2-Trichloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1-Dichloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1-Dichloroethene
GC/MS	EPA 8260B/C; EPA 524.2	1, 1-Dichloropropene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B/C	1,2,3-Trimethylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 4-Trimethylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dibromoethane (EDB)
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 2-Dichlorobenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 2-Dichloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 2-Dichloropropane
GC/MS	EPA 8260B/C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 3, 5-Trimethylbenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 3-Dichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 3-Dichloropropane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 4-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 4-Dioxane
GC/MS	EPA 8260B/C	1-Chlorohexane
GC/MS	EPA 8260B/C; EPA 524.2	2, 2-Dichloropropane
GC/MS	EPA 8260B/C; EPA 524.2	2-Butanone
GC/MS	EPA 624; EPA 8260B/C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/C; EPA 524.2	2-Chlorotoluene
GC/MS	EPA 8260B/C; EPA 524.2	2-Hexanone
GC/MS	EPA 8260B/C; EPA 524.2	4-Chlorotoluene
GC/MS	EPA 8260B/C; EPA 524.2	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C; EPA 524.2	Acetone
GC/MS	EPA 8260B/C	Acetonitrile
GC/MS	EPA 624; EPA 8260B/C	Acrolein
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Acrylonitrile
GC/MS	EPA 8260B/C; EPA 524.2	Allyl chloride
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Benzene
GC/MS	EPA 8260B/C	Benzyl chloride
GC/MS	EPA 8260B/C; EPA 524.2	Bromobenzene
GC/MS	EPA 8260B/C; EPA 524.2	Bromochloromethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Bromodichloromethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Bromoform
GC/MS	EPA 8260B/C; EPA 524.2	Carbon disulfide
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Carbon tetrachloride
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Chlorobenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Chloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Chloroform
GC/MS	EPA 8260B/C	Chloroprene
GC/MS	EPA 8260B/C; EPA 524.2	cis-1, 2-Dichloroethene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B/C	Cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260B/C	Cyclohexane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Dibromochloromethane
GC/MS	EPA 8260B/C; EPA 524.2	Dibromomethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Dichlorodifluoromethane
GC/MS	EPA 8260B/C; EPA 524.2	Diethyl ether
GC/MS	EPA 8260B/C	Di-isopropylether
GC/MS	EPA 8260B/C; EPA 524.2	Ethyl methacrylate
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Ethylbenzene
GC/MS	EPA 8260B/C	Ethyl-t-butylether
GC/MS	EPA 8260B/C; EPA 524.2	Hexachlorobutadiene
GC/MS	EPA 8260B/C	Iodomethane
GC/MS	EPA 8260B/C	Isobutyl alcohol
GC/MS	EPA 8260B/C	Isopropyl alcohol
GC/MS	EPA 8260B/C; EPA 524.2	Isopropyl benzene
GC/MS	EPA 8260B/C; EPA 524.2	m p-xylenes
GC/MS	EPA 8260B/C	Methyl acetate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C; EPA 524.2	Methacrylonitrile
GC/MS	EPA 624 / 8260B,C	Methyl bromide (Bromomethane)
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B/C; EPA 524.2	Methyl methacrylate
GC/MS	EPA 8260B/C; EPA 524.2	Methyl tert-butyl ether
GC/MS	EPA 8260B/C	Methylcyclohexane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Methylene chloride
GC/MS	EPA 8260B/C; EPA 524.2	Naphthalene
GC/MS	EPA 8260B/C; EPA 524.2	n-Butylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	n-Propylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	o-Xylene
GC/MS	EPA 8260B/C	Pentachloroethane
GC/MS	EPA 8260B/C; EPA 524.2	p-Isopropyltoluene
GC/MS	EPA 8260B/C; EPA 524.2	Propionitrile
GC/MS	EPA 8260B/C; EPA 524.2	sec-butylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	Styrene
GC/MS	EPA 8260B/C	t-Amylmethylether
GC/MS	EPA 8260B/C; EPA 524.2	tert-Butyl alcohol
GC/MS	EPA 8260B/C	tert-Butylbenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	EPA 8260B/C; EPA 524.2	Tetrahydrofuran
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Toluene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	trans-1, 2-Dichloroethylene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B/C; EPA 524.2	trans-1, 4-Dichloro-2-butuene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Trichloroethene (Trichloroethylene)
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Trichlorofluoromethane
GC/MS	EPA 8260B/C	Vinyl acetate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Vinyl chloride
GC/MS	EPA 624 / 8260B,C	Xylene
GC/MS	EPA 8260B/C SIM	1,1,1,2-Tetrachloroethane
GC/MS	8260B, C SIM	1,1,1-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/C SIM	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,2,3-Trichloropropane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethene
GC/MS	EPA 8260B/C SIM	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/C SIM	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C SIM	1,2-Dibromoethane
GC/MS	EPA 8260B/C SIM	1,2-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,2-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,3-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,3-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,4-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	2-Hexanone
GC/MS	EPA 8260B/C SIM	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C SIM	Benzene
GC/MS	EPA 8260B/C SIM	Bromodichloromethane
GC/MS	EPA 8260B/C SIM	Carbon Tetrachloride
GC/MS	EPA 8260B/C SIM	Chloroform
GC/MS	EPA 8260B/C SIM	Chloromethane
GC/MS	EPA 8260B/C SIM	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Dibromochloromethane
GC/MS	EPA 8260B/C SIM	Ethylbenzene
GC/MS	EPA 8260B/C SIM	Isopropylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C SIM	Hexachlorobutadiene
GC/MS	EPA 8260B/C SIM	Methylcyclohexane
GC/MS	EPA 8260B/C SIM	m,p-Xylene
GC/MS	EPA 8260B/C SIM	o-Xylene
GC/MS	EPA 8260B/C SIM	Tetrachloroethene
GC/MS	EPA 8260B/C SIM	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	Trans-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Trichloroethene
GC/MS	EPA 8260B/C SIM	Trichlorofluoromethane
GC/MS	EPA 8260B/C SIM	Vinyl Chloride
GC/MS	EPA 8260B/C SIM	Xylenes (total)
GC/MS	EPA 8270C/D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1, 3, 5-Trinitrobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 3-Dinitrobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 4-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 4-Dioxane
GC/MS	EPA 8270C/D	1, 4-Naphthoquinone
GC/MS	EPA 8270C/D	1, 4-Phenylenediamine
GC/MS	EPA 8270C/D	1-Chloronaphthalene
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	1-Naphthylamine
GC/MS	EPA 8270C/D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2, 4, 5-Trochlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 4, 6-Trichlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dichlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dimethylphenol
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dinitrophenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dinitrotoluene (2, 4-DNT)
GC/MS	EPA 8270C/D	2, 6-Dichlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 6-Dinitrotoluene (2, 6-DNT)
GC/MS	EPA 8270C/D	2-Acetylaminofluorene
GC/MS	EPA 625; EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 625; EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 625; EPA 8270C/D	2-Methyl-4 6-dinitrophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol
GC/MS	EPA 8270C/D	2-Naphthylamine
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 625; EPA 8270C/D	2-Nitrophenol
GC/MS	EPA 8270C/D	2-Picoline
GC/MS	EPA 8270C/D	3-Methylcholanthrene
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4-Aminobiphenyl
GC/MS	EPA 625; EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 625; EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 625; EPA 8270C/D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C/D	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270C/D	3, 4-Methylphenol
GC/MS	EPA 8270C/D	4-Nitroaniline
GC/MS	EPA 625; EPA 8270C/D	4-Nitrophenol
GC/MS	EPA 8270C/D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	5-Nitro-o-toluidine
GC/MS	EPA 8270C/D	7, 12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270C/D	a a-Dimethylphenethylamine
GC/MS	EPA 625; EPA 8270C/D	Acenaphthene
GC/MS	EPA 625; EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetophenone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 625; EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Aramite
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Azobenzene
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 625; EPA 8270C/D	Benzidine
GC/MS	EPA 625; EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 625; EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 625; EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 625; EPA 8270C/D	Benzo(g h i)perylene
GC/MS	EPA 625; EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	1,1-Biphenyl
GC/MS	EPA 625; EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 625; EPA 8270C/D	bis(2-Chloroethyl) ether
GC/MS	EPA 625; EPA 8270C/D	bis(2-Chloroisopropyl) ether (2, 2`-Oxybis(1-chloropropane)
GC/MS	EPA 625; EPA 8270C/D	bis(2-Ethylhexyl)adipate
GC/MS	EPA 625; EPA 8270C/D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 625; EPA 8270C/D	Butyl benzyl phthalate
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chlorobenzilate
GC/MS	EPA 625; EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Diallate
GC/MS	EPA 8270C/D	Dibenzo(a,j)acridine
GC/MS	EPA 625; EPA 8270C/D	Dibenz(a h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran
GC/MS	EPA 8270C/D	Diethyladipate
GC/MS	EPA 625; EPA 8270C/D	Diethyl phthalate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Dimethoate
GC/MS	EPA 625; EPA 8270C/D	Dimethyl phthalate
GC/MS	EPA 625; EPA 8270C/D	Di-n-butyl phthalate
GC/MS	EPA 625; EPA 8270C/D	Di-n-octyl phthalate
GC/MS	EPA 8270C/D	Dinoseb
GC/MS	EPA 8270C/D	Disulfoton
GC/MS	EPA 8270C/D	Ethyl methanesulfonate
GC/MS	EPA 8270C/D	Ethyl parathion
GC/MS	EPA 8270C/D	Ethyl methacrylate
GC/MS	EPA 8270C/D	Famfur
GC/MS	EPA 625; EPA 8270C/D	Fluoranthene
GC/MS	EPA 625; EPA 8270C/D	Fluorene
GC/MS	EPA 625; EPA 8270C/D	Hexachlorobenzene
GC/MS	EPA 625; EPA 8270C/D	Hexachlorobutadiene
GC/MS	EPA 625; EPA 8270C/D	Hexachlorocyclopentadiene
GC/MS	EPA 625; EPA 8270C/D	Hexachloroethane
GC/MS	EPA 8270C/D	Hexachlorophene
GC/MS	EPA 8270C/D	Hexachloropropene
GC/MS	EPA 625; EPA 8270C/D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C/D	Isodrin
GC/MS	EPA 625; EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Isosafrole
GC/MS	EPA 8270C/D	Kepone
GC/MS	EPA 8270C/D	Methapyriline
GC/MS	EPA 8270C/D	Methy methanesulfonate
GC/MS	EPA 8270C/D	Methyl parathion
GC/MS	EPA 625; EPA 8270C/D	Naphthalene
GC/MS	EPA 625; EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	n-Nitrosodiethylamine
GC/MS	EPA 625; EPA 8270C/D	n-Nitrosodimethylamine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	n-Nitroso-di-n-butylamine
GC/MS	EPA 625; EPA 8270C/D	n-Nitrosodi-n-propylamine
GC/MS	EPA 625; EPA 8270C/D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C/D	n-Nitrosomorpholine
GC/MS	EPA 8270C/D	n-Nitrosopiperidine
GC/MS	EPA 8270C/D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C/D	O,O,O-Triethyl phosphorothioate
GC/MS	EPA 8270C/D	o,o-Diethyl o-2pyrazinyl phosphorothioate
GC/MS	EPA 8270C/D	o-Toluidine
GC/MS	EPA 8270C/D	Pentachlorobenzene
GC/MS	EPA 8270C/D	Pentachloronitrobenzene
GC/MS	EPA 625; EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenacetin
GC/MS	EPA 625; EPA 8270C/D	Phenanthrene
GC/MS	EPA 625; EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Phorate
GC/MS	EPA 8270C/D	Pronamide
GC/MS	EPA 625; EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/MS	EPA 8270C/D	Safrole
GC/MS	EPA 8270C/D	Sulfotepp
GC/MS	EPA 8270C/D	Thionazin
GC/MS	EPA 625; EPA 8270C/D	3, 3’-Dichlorobenzidine
GC/MS	EPA 8270C/D	3, 3’-Dimethylbenzidine
GC/MS	EPA 8270C/D SIM	1,1'-Biphenyl
GC/MS	EPA 8270C/D SIM	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D SIM	1,4-Dioxane
GC/MS	EPA 8270C/D SIM	1-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2,2'-Oxybis(1-chloropropane
GC/MS	EPA 8270C/D SIM	2,3,4,6-Tetrachlorophenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dimethylphenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrophenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2-Chloronaphthalene
GC/MS	EPA 8270C/D SIM	2-Chlorophenol
GC/MS	EPA 8270C/D SIM	2-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2-Methylphenol
GC/MS	EPA 8270C/D SIM	2-Nitroaniline
GC/MS	EPA 8270C/D SIM	2-Nitrophenol
GC/MS	EPA 8270C/D SIM	3&4-Methylphenol
GC/MS	EPA 8270C/D SIM	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/D SIM	3-Nitroaniline
GC/MS	EPA 8270C/D SIM	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/D SIM	4-Bromophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D SIM	4-Chloroaniline
GC/MS	EPA 8270C/D SIM	4-Chlorophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Nitroaniline
GC/MS	EPA 8270C/D SIM	4-Nitrophenol
GC/MS	EPA 8270C/D SIM	Acenaphthene
GC/MS	EPA 8270C/D SIM	Acenaphthylene
GC/MS	EPA 8270C/D SIM	Acetophenone
GC/MS	EPA 8270C/D SIM	Anthracene
GC/MS	EPA 8270C/D SIM	Atrazine
GC/MS	EPA 8270C/D SIM	Benzaldehyde
GC/MS	EPA 8270C/D SIM	Benzo(a)anthracene
GC/MS	EPA 8270C/D SIM	Benzo(a)pyrene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/D SIM	Bis(2-ethylhexyl)phthalate
GC/MS	EPA 8270C/D SIM	Butylbenzylphthalate
GC/MS	EPA 8270C/D SIM	Caprolactam
GC/MS	EPA 8270C/D SIM	Carbazole
GC/MS	EPA 8270C/D SIM	Chrysene
GC/MS	EPA 8270C/D SIM	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D SIM	Dibenzofuran
GC/MS	EPA 8270C/D SIM	Diethylphthalate
GC/MS	EPA 8270C/D SIM	Dimethyl phthalate
GC/MS	EPA 8270C/D SIM	Di-n-butylphthalate
GC/MS	EPA 8270C/D SIM	Di-n-octylphthalate
GC/MS	EPA 8270C/D SIM	Fluoranthene
GC/MS	EPA 8270C/D SIM	Fluorene
GC/MS	EPA 8270C/D SIM	Hexachlorobenzene
GC/MS	EPA 8270C/D SIM	Hexachlorobutadiene
GC/MS	EPA 8270C/D SIM	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D SIM	Hexachloroethane
GC/MS	EPA 8270C/D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D SIM	Isophorone
GC/MS	EPA 8270C/D SIM	Naphthalene
GC/MS	EPA 8270C/D SIM	Nitrobenzene
GC/MS	EPA 8270C/D SIM	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270C/D SIM	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D SIM	Pentachlorophenol
GC/MS	EPA 8270C/D SIM	Phenanthrene
GC/MS	EPA 8270C/D SIM	Phenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Pyrene
HPLC/UV	EPA 8330A/B	1, 3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A/B	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330A/B	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A/B	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Amino-4, 6 -Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Nitrotoluene
HPLC/UV	EPA 8330A/B	3-Nitrotoluene
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	4-Amino-2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	4-Nitrotoluene
HPLC/UV	EPA 8330A/B	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A/B	Hexahydro-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A/B	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330A/B	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/B	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A/B	Tetryl
CVAA	EPA 245.1; EPA 7470A	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 200.7; EPA 6010B/C	Aluminum
ICP/AES	EPA 200.7; EPA 6010B/C	Antimony
ICP/AES	EPA 200.7; EPA 6010B/C	Arsenic
ICP/AES	EPA 200.7; EPA 6010B/C	Barium
ICP/AES	EPA 200.7; EPA 6010B/C	Beryllium
ICP/AES	EPA 200.7; EPA 6010B/C	Boron
ICP/AES	EPA 200.7; EPA 6010B/C	Cadmium
ICP/AES	EPA 200.7; EPA 6010B/C	Calcium
ICP/AES	EPA 200.7; EPA 6010B/C	Chromium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/AES	EPA 200.7; EPA 6010B/C	Cobalt
ICP/AES	EPA 200.7; EPA 6010B/C	Copper
ICP/AES	EPA 200.7; EPA 6010B/C	Iron
ICP/AES	EPA 200.7; EPA 6010B/C	Lead
ICP/AES	EPA 200.7; EPA 6010B/C	Magnesium
ICP/AES	EPA 200.7; EPA 6010B/C	Manganese
ICP/AES	EPA 200.7; EPA 6010B/C	Molybdenum
ICP/AES	EPA 200.7; EPA 6010B/C	Nickel
ICP/AES	EPA 200.7; EPA 6010B/C	Potassium
ICP/AES	EPA 200.7; EPA 6010B/C	Selenium
ICP/AES	EPA 200.7; EPA 6010B/C	Silicon
ICP/AES	EPA 200.7; EPA 6010B/C	Silver
ICP/AES	EPA 200.7; EPA 6010B/C	Sodium
ICP/AES	EPA 6010B/C	Strontium
ICP/AES	EPA 200.7; EPA 6010B/C	Thallium
ICP/AES	EPA 200.7; EPA 6010B/C	Tin
ICP/AES	EPA 200.7; EPA 6010B/C	Titanium
ICP/AES	EPA 200.7; EPA 6010B/C	Vanadium
ICP/AES	EPA 200.7; EPA 6010B/C	Zinc
ICP/MS	EPA 200.8; EPA 6020A	Aluminum
ICP/MS	EPA 200.8; EPA 6020A	Antimony
ICP/MS	EPA 200.8; EPA 6020A	Arsenic
ICP/MS	EPA 200.8; EPA 6020A	Barium
ICP/MS	EPA 200.8; EPA 6020A	Beryllium
ICP/MS	EPA 200.8; EPA 6020A	Boron
ICP/MS	EPA 200.8; EPA 6020A	Cadmium
ICP/MS	EPA 200.8; EPA 6020A	Calcium
ICP/MS	EPA 200.8; EPA 6020A	Chromium
ICP/MS	EPA 200.8; EPA 6020A	Cobalt
ICP/MS	EPA 200.8; EPA 6020A	Copper
ICP/MS	EPA 200.8; EPA 6020A	Iron

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 200.8; EPA 6020A	Lead
ICP/MS	EPA 200.8; EPA 6020A	Magnesium
ICP/MS	EPA 200.8; EPA 6020A	Manganese
ICP/MS	EPA 200.8; EPA 6020A	Molybdenum
ICP/MS	EPA 200.8; EPA 6020A	Nickel
ICP/MS	EPA 200.8; EPA 6020A	Potassium
ICP/MS	EPA 200.8; EPA 6020A	Selenium
ICP/MS	EPA 200.8; EPA 6020A	Silicon
ICP/MS	EPA 200.8; EPA 6020A	Silver
ICP/MS	EPA 200.8; EPA 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 200.8; EPA 6020A	Thallium
ICP/MS	EPA 200.8; EPA 6020A	Tin
ICP/MS	EPA 200.8; EPA 6020A	Titanium
ICP/MS	EPA 200.8; EPA 6020A	Tungsten
ICP/MS	EPA 200.8	Uranium
ICP/MS	EPA 200.8; EPA 6020A	Vanadium
ICP/MS	EPA 200.8; EPA 6020A	Zinc
IC	EPA 300.0; EPA 9056A	Bromide
IC	EPA 300.0; EPA 9056A	Chloride
IC	EPA 300.0; EPA 9056A	Fluoride
IC	EPA 300.0; EPA 9056A	Nitrate as N
IC	EPA 300.0; EPA 9056A	Nitrite as N
IC	EPA 300.0; EPA 9056A	Nitrate + Nitrite
IC	EPA 300.0; EPA 9056A	Orthophosphate as P
IC	EPA 300.0; EPA 9056A	Sulfate
IC	SOP CA-776	Lactic Acid
IC	SOP CA-776	Acetic Acid
IC	SOP CA-776	Propionic Acid
IC	SOP CA-776	Formic Acid
IC	SOP CA-776	Butyric Acid

Non-Potable Water		
Technology	**Method**	**Analyte**
IC	SOP CA-776	Pyruvic Acid
IC	SOP CA-776	i-Pentanoic Acid
IC	SOP CA-776	Pentanoic Acid
IC	SOP CA-776	i-Hexanoic Acid
IC	SOP CA-776	Hexanoic Acid
Titration	EPA 310.1; SM 2320B	Alkalinity
Caculation	SM 2340B	Hardness
Gravimetric	EPA 1664A; EPA 9070A	Oil and Grease, Oil and Grease with SGT
Gravimetric	SM 2540B/C/D	Solids
ISE	EPA 120.1; SM 2510B	Conductivity
ISE	SM 2520B	Practical Salinity
ISE	SM 4500F- C	Fluoride
ISE	SM 4500H+ B	pH
ISE	SM 5210B	TBOD / CBOD
Physical	EPA 1010A	Ignitability
Physical	EPA 9040C	pH
Titration	SM 2340C	Hardness
Titration	SM 4500SO_3 B	Sulfite
Titration	EPA 9034; SM 4500S^{2-} F	Sulfide
Titration	EPA SW-846 Chapter 7.3.4	Reactive Sulfide
IR	EPA 9060A; SM 5310B	Total organic carbon
Turbidimetric	EPA 180.1; SM 2130B	Turbidity
Turbidimetric	EPA 9038; ASTM 516-02	Sulfate
UV/VIS	EPA 335.4; EPA 9012B; SM 4500-CN G	Amenable cyanide
UV/VIS	EPA 350.1; SM 4500NH3 H	Ammonia as N
UV/VIS	SM 3500Fe D	Ferrous Iron
UV/VIS	EPA 351.2	Kjeldahl nitrogen - total
UV/VIS	EPA 353.2; SM 4500NO3 F	Nitrate + Nitrite
UV/VIS	EPA 353.2; SM 4500NO3 F	Nitrate as N
UV/VIS	EPA 353.2; SM 4500NO3 F	Nitrite as N
UV/VIS	EPA 365.2; SM 4500P E	Orthophosphate as P

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/VIS	EPA 365.4	Phosphorus total
UV/VIS	EPA 821/R-91-100	AVS-SEM
UV/VIS	EPA 410.4	COD
UV/VIS	EPA 420.1; EPA 9065	Total Phenolics
UV/VIS	SM 4500Cl G	Total Residual Chlorine
UV/VIS	SM 5540C	MBAS
UV/VIS	EPA 7196A; SM 3500-Cr D	Chromium VI
UV/VIS	EPA 9012B; EPA 335.4	Total Cyanide
UV/VIS	EPA 9251; SM 4500Cl E	Chloride
UV/VIS	EPA SW-846 Chapter 7.3.4	Reactive Cyanide

Preparation	Method	Type
Cleanup Methods	EPA 3640A	Gel Permeation Clean-up
Cleanup Methods	EPA 3630C	Silica Gel
Cleanup Methods	EPA 3660B	Sulfur Clean-Up
Cleanup Methods	EPA 3665A	Sulfuric Acid Clean-Up
Organic Preparation	EPA 3510C	Separatory Funnel Extraction
Organic Preparation	EPA 3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	EPA 3010A	Hotblock
Volatile Organic Preparation	EPA 5030C	Purge and Trap

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	2,4`-DDD
GC/ECD	EPA 8081B	2,4`-DDE
GC/ECD	EPA 8081B	2,4`-DDT
GC/ECD	EPA 8081B	4, 4`-DDD
GC/ECD	EPA 8081B	4, 4`-DDE
GC/ECD	EPA 8081B	4, 4`-DDT
GC/ECD	EPA 8081B	Aldrin

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 608; EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 8081B	Cis-Nonachlor
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Hexachlorobenzene
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Oxychlordane
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8081B	Trans-Nonachlor
GC/ECD	EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 5', 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4, 5-Tetrachlorobiphenyl (BZ 48)
GC/ECD	EPA 8082A	2, 2', 4, 5'-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5'-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3, 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015C/D	Diesel range organics (DRO)
GC/FID	EPA 8015C/D	Total Petroleum Hydrocarbons (TPH)
GC/FID	EPA 8015C/D	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	MA DEP EPH EPA 3546	Extractable Petroleum Hydrocarbons Microwave Extraction Preparation
GC/FID	CT-ETPH	Total Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics
GC/ECD	EPA 8011	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/C	1, 1, 1-Trichloroethane
GC/MS	EPA 8260B/C	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B/C	1, 1-Dichloroethane

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	1, 1-Dichloroethylene
GC/MS	EPA 8260B/C	1, 1-Dichloropropene
GC/MS	EPA 8260B/C	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B/C	1,2,3-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 2, 4-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C	1, 2-Dibromoethane
GC/MS	EPA 8260B/C	1, 2-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 2-Dichloroethane
GC/MS	EPA 8260B/C	1, 2-Dichloropropane
GC/MS	EPA 8260B/C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 3, 5-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 3-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 3-Dichloropropane
GC/MS	EPA 8260B/C	1, 4-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 4-Dioxane
GC/MS	EPA 8260B/C	1-Chlorohexane
GC/MS	EPA 8260B/C	2, 2-Dichloropropane
GC/MS	EPA 8260B/C	2-Butanone
GC/MS	EPA 8260B/C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/C	2-Chlorotoluene
GC/MS	EPA 8260B/C	2-Hexanone
GC/MS	EPA 8260B/C	4-Chlorotoluene
GC/MS	EPA 8260B/C	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C	Acetone
GC/MS	EPA 8260B/C	Acetonitrile
GC/MS	EPA 8260B/C	Acrolein
GC/MS	EPA 8260B/C	Acrylonitrile
GC/MS	EPA 8260B/C	Allyl chloride

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	Benzene
GC/MS	EPA 8260B/C	Benzyl chloride
GC/MS	EPA 8260B/C	Bromobenzene
GC/MS	EPA 8260B/C	Bromochloromethane
GC/MS	EPA 8260B/C	Bromodichloromethane
GC/MS	EPA 8260B/C	Bromoform
GC/MS	EPA 8260B/C	Carbon disulfide
GC/MS	EPA 8260B/C	Carbon tetrachloride
GC/MS	EPA 8260B/C	Chlorobenzene
GC/MS	EPA 8260B/C	Chloroethane
GC/MS	EPA 8260B/C	Chloroform
GC/MS	EPA 8260B/C	Chloroprene
GC/MS	EPA 8260B/C	cis-1, 2-Dichloroethene
GC/MS	EPA 8260B/C	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B/C	cis-1,3-Dichloro-2-butene
GC/MS	EPA 8260B/C	Cyclohexane
GC/MS	EPA 8260B/C	Dibromochloromethane
GC/MS	EPA 8260B/C	Dibromomethane
GC/MS	EPA 8260B/C	Dichlorodifluoromethane
GC/MS	EPA 8260B/C	Diethyl ether
GC/MS	EPA 8260B/C	Di-isopropylether
GC/MS	EPA 8260B/C	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B/C	Ethyl methacrylate
GC/MS	EPA 8260B/C	Ethylbenzene
GC/MS	EPA 8260B/C	Ethyl-t-butylether
GC/MS	EPA 8260B/C	Hexachlorobutadiene
GC/MS	EPA 8260B/C	Iodomethane
GC/MS	EPA 8260B/C	Isobutyl alcohol
GC/MS	EPA 8260B/C	Isopropyl alcohol
GC/MS	EPA 8260B/C	Isopropyl benzene
GC/MS	EPA 8260B/C	m p-xylenes

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B, C	Methyl acetate
GC/MS	EPA 8260B/C	Methacrylonitrile
GC/MS	EPA 8260B/C	Methyl bromide (Bromomethane)
GC/MS	EPA 8260B/C	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B/C	Methyl methacrylate
GC/MS	EPA 8260B/C	Methyl tert-butyl ether
GC/MS	EPA 8260B/C	Methylcyclohexane
GC/MS	EPA 8260B/C	Methylene chloride
GC/MS	EPA 8260B/C	Naphthalene
GC/MS	EPA 8260B/C	n-Butylbenzene
GC/MS	EPA 8260B/C	n-proplybenzene
GC/MS	EPA 8260B/C	o-Xylene
GC/MS	EPA 8260B/C	pentachloroethane
GC/MS	EPA 8260B/C	p-Isopropyltoluene
GC/MS	EPA 8260B/C	Propionitrile
GC/MS	EPA 8260B/C	sec-butylbenzene
GC/MS	EPA 8260B/C	Styrene
GC/MS	EPA 8260B/C	t-Amylmethylether
GC/MS	EPA 8260B/C	tert-Butyl alcohol
GC/MS	EPA 8260B/C	tert-Butylbenzene
GC/MS	EPA 8260B/C	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260B/C	Tetrahydrofuran
GC/MS	EPA 8260B/C	Toluene
GC/MS	EPA 8260B/C	trans-1, 2-Dichloroethylene
GC/MS	EPA 8260B/C	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B/C	Trans-1, 4-Dichloro-2-butuene
GC/MS	EPA 8260B/C	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260B/C	Trichlorofluoromethane
GC/MS	EPA 8260B/C	Vinyl acetate
GC/MS	EPA 8260B/C	Vinyl chloride
GC/MS	EPA 8260B/C	Xylene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C SIM	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/C SIM	1,1,1-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/C SIM	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,2,3-Trichloropropane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethene
GC/MS	EPA 8260B/C SIM	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/C SIM	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C SIM	1,2-Dibromoethane
GC/MS	EPA 8260B/C SIM	1,2-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,2-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,3-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,3-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,4-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	2-Hexanone
GC/MS	EPA 8260B/C SIM	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C SIM	Benzene
GC/MS	EPA 8260B/C SIM	Bromodichloromethane
GC/MS	EPA 8260B/C SIM	Carbon Tetrachloride
GC/MS	EPA 8260B/C SIM	Chloroform
GC/MS	EPA 8260B/C SIM	Chloromethane
GC/MS	EPA 8260B/C SIM	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Dibromochloromethane
GC/MS	EPA 8260B/C SIM	Ethylbenzene
GC/MS	EPA 8260B/C SIM	Isopropylbenzene
GC/MS	EPA 8260B/C SIM	Hexachlorobutadiene
GC/MS	EPA 8260B/C SIM	Methylcyclohexane

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C SIM	m,p-Xylene
GC/MS	EPA 8260B/C SIM	o-Xylene
GC/MS	EPA 8260B/C SIM	Tetrachloroethene
GC/MS	EPA 8260B/C SIM	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	Trans-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Trichloroethene
GC/MS	EPA 8260B/C SIM	Trichlorofluoromethane
GC/MS	EPA 8260B/C SIM	Vinyl Chloride
GC/MS	EPA 8260B/C SIM	Xylenes (total)
GC/MS	EPA 8270C/D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1, 3, 5-Trinitrobenzene
GC/MS	EPA 8270C/D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 3-Dinitrobenzene
GC/MS	EPA 8270C/D	1, 4-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 4-Dioxane
GC/MS	EPA 8270C/D	1, 4-Naphthoquinone
GC/MS	EPA 8270C/D	1, 4-Phenylenediamine
GC/MS	EPA 8270C/D	1,1-Biphenyl
GC/MS	EPA 8270C/D	1-Chloronaphthalene
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	1-Naphthylamine
GC/MS	EPA 8270C/D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2, 4, 5-Trochlorophenol
GC/MS	EPA 8270C/D	2, 4, 6-Trichlorophenol
GC/MS	EPA 8270C/D	2, 4-Dichlorophenol
GC/MS	EPA 8270C/D	2, 4-Dimethylphenol
GC/MS	EPA 8270C/D	2, 4-Dinitrophenol
GC/MS	EPA 8270C/D	2, 4-Dinitrotoluene (2 4-DNT)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	2, 6-Dichlorophenol
GC/MS	EPA 8270C/D	2, 6-Dinitrotoluene (2 6-DNT)
GC/MS	EPA 8270C/D	2-Acetylaminofluorene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methyl-4, 6-dinitrophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol
GC/MS	EPA 8270C/D	2-Naphthylamine
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol
GC/MS	EPA 8270C/D	2-Picoline
GC/MS	EPA 8270C/D	3, 3`-Dichlorobenzidine
GC/MS	EPA 8270C/D	3, 3'-Dimethylbenzidine
GC/MS	EPA 8270C/D	3,4-Methylphenol
GC/MS	EPA 8270C/D	3-Methylcholanthrene
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4-Aminobiphenyl
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C/D	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270C/D	4-Nitroaniline
GC/MS	EPA 8270C/D	4-Nitrophenol
GC/MS	EPA 8270C/D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	5-Nitro-o-toluidine
GC/MS	EPA 8270C/D	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270C/D	a a-Dimethylphenethylamine
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Acetophenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Aramite
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Azobenzene
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g h i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270C/D	bis(2-Chloroisopropyl) ether (2, 2`-Oxybis(1-chloropropane))
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 625; EPA 8270C/D	Bis(2-Ethylhexyl)adipate
GC/MS	EPA 8270C/D	Butyl benzyl phthalate
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chlorobenzilate
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Diallate
GC/MS	EPA 8270C/D	Dibenz(a h)anthracene
GC/MS	EPA 8270C/D	Dibenzo(a,j)acridine
GC/MS	EPA 8270C/D	Dibenzofuran
GC/MS	EPA 8270C/D	Diethyl phthalate
GC/MS	EPA 8270C/D	Diethyladipate

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Dimethoate
GC/MS	EPA 8270C/D	Dimethyl phthalate
GC/MS	EPA 8270C/D	Di-n-butyl phthalate
GC/MS	EPA 8270C/D	Di-n-octyl phthalate
GC/MS	EPA 8270C/D	Dinoseb
GC/MS	EPA 8270C/D	Disulfoton
GC/MS	EPA 8270C/D	Ethyl methacrylate
GC/MS	EPA 8270C/D	Ethyl methanesulfonate
GC/MS	EPA 8270C/D	Ethyl parathion
GC/MS	EPA 8270C/D	Famfur
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene
GC/MS	EPA 8270C/D	Hexachlorobutadiene
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D	Hexachloroethane
GC/MS	EPA 8270C/D	Hexachlorophene
GC/MS	EPA 8270C/D	Hexachloropropene
GC/MS	EPA 8270C/D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C/D	Isodrin
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Isosafrole
GC/MS	EPA 8270C/D	Kepone
GC/MS	EPA 8270C/D	Methapyriline
GC/MS	EPA 8270C/D	Methyl methanesulfonate
GC/MS	EPA 8270C/D	Methyl parathion
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	n-Nitrosodiethylamine
GC/MS	EPA 8270C/D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/D	n-Nitroso-di-n-butylamine

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C/D	n-Nitrosomorpholine
GC/MS	EPA 8270C/D	n-Nitrosopiperidine
GC/MS	EPA 8270C/D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C/D	O, O, O-Triethyl phosphorothioate
GC/MS	EPA 8270C/D	o,o-Diethyl o-2-pyrazinyl phosphorothioate
GC/MS	EPA 8270C/D	o-Toluidine
GC/MS	EPA 8270C/D	Pentachlorobenzene
GC/MS	EPA 8270C/D	Pentachloronitrobenzene
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenacetin
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Phorate
GC/MS	EPA 8270C/D	Pronamide
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/MS	EPA 8270C/D	Safrole
GC/MS	EPA 8270C/D	Sulfotepp
GC/MS	EPA 8270C/D	Thionazin
GC/MS	EPA 8270C/D SIM	1,1'-Biphenyl
GC/MS	EPA 8270C/D SIM	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D SIM	1,4-Dioxane
GC/MS	EPA 8270C/D SIM	1-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2,2'-Oxybis(1-chloropropane
GC/MS	EPA 8270C/D SIM	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D SIM	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dichlorophenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	2,4-Dimethylphenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrophenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2-Chloronaphthalene
GC/MS	EPA 8270C/D SIM	2-Chlorophenol
GC/MS	EPA 8270C/D SIM	2-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2-Methylphenol
GC/MS	EPA 8270C/D SIM	2-Nitroaniline
GC/MS	EPA 8270C/D SIM	2-Nitrophenol
GC/MS	EPA 8270C/D SIM	3&4-Methylphenol
GC/MS	EPA 8270C/D SIM	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/D SIM	3-Nitroaniline
GC/MS	EPA 8270C/D SIM	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/D SIM	4-Bromophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D SIM	4-Chloroaniline
GC/MS	EPA 8270C/D SIM	4-Chlorophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Nitroaniline
GC/MS	EPA 8270C/D SIM	4-Nitrophenol
GC/MS	EPA 8270C/D SIM	Acenaphthene
GC/MS	EPA 8270C/D SIM	Acenaphthylene
GC/MS	EPA 8270C/D SIM	Acetophenone
GC/MS	EPA 8270C/D SIM	Anthracene
GC/MS	EPA 8270C/D SIM	Atrazine
GC/MS	EPA 8270C/D SIM	Benzaldehyde
GC/MS	EPA 8270C/D SIM	Benzo(a)anthracene
GC/MS	EPA 8270C/D SIM	Benzo(a)pyrene
GC/MS	EPA 8270C/D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/D SIM	Bis(2-ethylhexyl)phthalate
GC/MS	EPA 8270C/D SIM	Butylbenzylphthalate
GC/MS	EPA 8270C/D SIM	Caprolactam
GC/MS	EPA 8270C/D SIM	Carbazole
GC/MS	EPA 8270C/D SIM	Chrysene
GC/MS	EPA 8270C/D SIM	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D SIM	Dibenzofuran
GC/MS	EPA 8270C/D SIM	Diethylphthalate
GC/MS	EPA 8270C/D SIM	Dimethyl phthalate
GC/MS	EPA 8270C/D SIM	Di-n-butylphthalate
GC/MS	EPA 8270C/D SIM	Di-n-octylphthalate
GC/MS	EPA 8270C/D SIM	Fluoranthene
GC/MS	EPA 8270C/D SIM	Fluorene
GC/MS	EPA 8270C/D SIM	Hexachlorobenzene
GC/MS	EPA 8270C/D SIM	Hexachlorobutadiene
GC/MS	EPA 8270C/D SIM	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D SIM	Hexachloroethane
GC/MS	EPA 8270C/D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D SIM	Isophorone
GC/MS	EPA 8270C/D SIM	Naphthalene
GC/MS	EPA 8270C/D SIM	Nitrobenzene
GC/MS	EPA 8270C/D SIM	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270C/D SIM	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D SIM	Pentachlorophenol
GC/MS	EPA 8270C/D SIM	Phenanthrene
GC/MS	EPA 8270C/D SIM	Phenol
GC/MS	EPA 8270C/D SIM	Pyrene
HPLC/UV	EPA 8330A	1 ,3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A	1, 3-Dinitrobenzene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4, 6-dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	3,5-Dinitroaniline
HPLC/UV	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene
HPLC/UV	EPA 8330A	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A	Hexahydr-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A	Tetryl
HPLC/UV	8330B (W/O Soil Grinding)	1, 3, 5-Trinitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	1, 3-Dinitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	2, 4, 6-Trinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2, 4-Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2, 6-Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2-Amino-4, 6 –Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	3-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	3,5-Dinitroaniline
HPLC/UV	8330B (W/O Soil Grinding)	4-Amino-2,3-Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	4-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	Ethylene glycol dinitrate (EGDN)
HPLC/UV	8330B (W/O Soil Grinding)	Hexahydr-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	8330B (W/O Soil Grinding)	Nitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	Nitroglycerin

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
HPLC/UV	8330B (W/O Soil Grinding)	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	8330B (W/O Soil Grinding)	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	8330B (W/O Soil Grinding)	Tetryl
CVAA	EPA 7471B	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 6010B/C	Aluminum
ICP/AES	EPA 6010B/C	Antimony
ICP/AES	EPA 6010B/C	Arsenic
ICP/AES	EPA 6010B/C	Barium
ICP/AES	EPA 6010B/C	Beryllium
ICP/AES	EPA 6010B/C	Boron
ICP/AES	EPA 6010B/C	Cadmium
ICP/AES	EPA 6010B/C	Calcium
ICP/AES	EPA 6010B/C	Chromium
ICP/AES	EPA 6010B/C	Cobalt
ICP/AES	EPA 6010B/C	Copper
ICP/AES	EPA 6010B/C	Iron
ICP/AES	EPA 6010B/C	Lead
ICP/AES	EPA 6010B/C	Magnesium
ICP/AES	EPA 6010B/C	Manganese
ICP/AES	EPA 6010B/C	Molybdenum
ICP/AES	EPA 6010B/C	Nickel
ICP/AES	EPA 6010B/C	Potassium
ICP/AES	EPA 6010B/C	Selenium
ICP/AES	EPA 6010B/C	Silicon
ICP/AES	EPA 6010B/C	Silver
ICP/AES	EPA 6010B/C	Sodium
ICP/AES	EPA 6010B/C	Strontium
ICP/AES	EPA 6010B/C	Thallium
ICP/AES	EPA 6010B/C	Tin
ICP/AES	EPA 6010B/C	Titanium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/AES	EPA 6010B/C	Vanadium
ICP/AES	EPA 6010B/C	Zinc
ICP/MS	EPA 6020A	Aluminum
ICP/MS	EPA 6020A	Antimony
ICP/MS	EPA 6020A	Arsenic
ICP/MS	EPA 6020A	Barium
ICP/MS	EPA 6020A	Beryllium
ICP/MS	EPA 6020A	Boron
ICP/MS	EPA 6020A	Cadmium
ICP/MS	EPA 6020A	Calcium
ICP/MS	EPA 6020A	Chromium
ICP/MS	EPA 6020A	Cobalt
ICP/MS	EPA 6020A	Copper
ICP/MS	EPA 6020A	Iron
ICP/MS	EPA 6020A	Lead
ICP/MS	EPA 6020A	Magnesium
ICP/MS	EPA 6020A	Manganese
ICP/MS	EPA 6020A	Molybdenum
ICP/MS	EPA 6020A	Nickel
ICP/MS	EPA 6020A	Potassium
ICP/MS	EPA 6020A	Selenium
ICP/MS	EPA 6020A	Silver
ICP/MS	EPA 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 6020A	Thallium
ICP/MS	EPA 6020A	Tin
ICP/MS	EPA 6020A	Titanium
ICP/MS	EPA 6020A	Tungsten
ICP/MS	EPA 6020A	Vanadium
ICP/MS	EPA 6020A	Zinc
IC	EPA 9056A	Chloride

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate as N
IC	EPA 9056A	Nitrite as N
IC	EPA 9056A	Orthophosphate
IC	EPA 9056A	Sulfate
Gravimetric	EPA 9071A; EPA 9071B	Oil and Grease, Oil and Grease with SGT
Physical	EPA 1010A	Ignitability
Physical	EPA 9045D	pH
Titration	EPA SW-846 Chapter 7.3.4	Reactive Sulfide
Titration	Walkley-Black	Total Organic Carbon
IR	Lloyd Kahn	Total organic carbon
Turbidimetric	EPA 9038; ASTM 516-02	Sulfate
UV/VIS	EPA 350.1; SM 4500NH3 H	Ammonia as N
UV/VIS	EPA 9251; SM 4500Cl E	Chloride
UV/VIS	EPA SW-846 Chapter 7.3.4	Reactive Cyanide
UV/VIS	EPA 821/R-91-100	AVS-SEM
UV/VIS	SM 3500Fe D	Ferrous Iron
Cleanup Methods	EPA 3630C	Silica Gel
UV/VIS	EPA 7196	Chromium VI
UV/VIS	EPA 7196A	Chromium VI
UV/VIS	EPA 9012B	Total cyanide
Preparation	**Method**	**Type**
Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	EPA 3660B	Sulfur Clean-up
Cleanup Methods	EPA 3620C	Florsil Clean-up
Cleanup Methods	EPA 3630C	Silica Gel Clean-up
Cleanup Methods	EPA 3640A	GPC Clean-up
Organic Preparation	EPA 3540C	Soxhlet Extraction
Organic Preparation	EPA 3545A	Pressurized Fluid Extraction

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
Organic Preparation	EPA 3546	Microwave Extraction Preparation for EPA 8082A, 8081B and 8270C, D
Organic Preparation	EPA 3550C	Sonication
Inorganics Preparation	EPA 3050B	Hotblock
Inorganics Preparation	EPA 3060A	Alkaline Digestion
Volatile Organics Preparation	EPA 5035/5035A	Closed System Purge and Trap

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	Propene
GC/MS	EPA TO-15	1, 1, 1-Trichloroethane
GC/MS	EPA TO-15	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA TO-15	1, 1, 2-Trichloroethane
GC/MS	EPA TO-15	1, 1-Dichloroethane
GC/MS	EPA TO-15	1, 1-Dichloroethylene
GC/MS	EPA TO-15	1, 2, 4-Trichlorobenzene
GC/MS	EPA TO-15	1, 2, 4-Trimethylbenzene
GC/MS	EPA TO-15	1, 2-Dibromoethane (EDB)
GC/MS	EPA TO-15	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
GC/MS	EPA TO-15	1, 2-Dichlorobenzene
GC/MS	EPA TO-15	1, 2-Dichloroethane
GC/MS	EPA TO-15	1, 2-Dichloroethenes (Total)
GC/MS	EPA TO-15	1, 2-Dichloropropane
GC/MS	EPA TO-15	1, 3, 5-Trimethylbenzene
GC/MS	EPA TO-15	1, 3-Butadiene
GC/MS	EPA TO-15	1, 3-Dichlorobenzene
GC/MS	EPA TO-15	1, 4-Dichlorobenzene
GC/MS	EPA TO-15	1,4-Difluorobenzene
GC/MS	EPA TO-15	1, 4-Dioxane
GC/MS	EPA TO-15	2-Butanone
GC/MS	EPA TO-15	2-Hexanone

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	2-Propanol
GC/MS	EPA TO-15	4-Ethyltoluene
GC/MS	EPA TO-15	4-Methyl-2-pentanone
GC/MS	EPA TO-15	Acetone
GC/MS	EPA TO-15	Acrolein
GC/MS	EPA TO-15	Benzene
GC/MS	EPA TO-15	Benzyl chloride
GC/MS	EPA TO-15	Bromochloromethane
GC/MS	EPA TO-15	Bromodichloromethane
GC/MS	EPA TO-15	Bromoform
GC/MS	EPA TO-15	Carbon disulfide
GC/MS	EPA TO-15	Carbon tetrachloride
GC/MS	EPA TO-15	Chlorobenzene
GC/MS	EPA TO-15	Chloroethane
GC/MS	EPA TO-15	Chloroform
GC/MS	EPA TO-15	Cis-1, 2-Dichloroethene
GC/MS	EPA TO-15	Cis-1, 3-Dichloropropene
GC/MS	EPA TO-15	Cyclohexane
GC/MS	EPA TO-15	Dibromochloromethane
GC/MS	EPA TO-15	Dichlorodifluoromethane (Freon 12)
GC/MS	EPA TO-15	Ethanol
GC/MS	EPA TO-15	Ethyl acetate
GC/MS	EPA TO-15	Ethylbenzene
GC/MS	EPA TO-15	Hexachlorobutadiene
GC/MS	EPA TO-15	Isopropyl alcohol
GC/MS	EPA TO-15	m, p-Xylene
GC/MS	EPA TO-15	Methyl bromide (Bromomethane)
GC/MS	EPA TO-15	Methyl chloride (Chloromethane)
GC/MS	EPA TO-15	Methyl methacrylate
GC/MS	EPA TO-15	Methyl tert-butyl ether
GC/MS	EPA TO-15	Methylene chloride

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	Naphthalene
GC/MS	EPA TO-15	n-Heptane
GC/MS	EPA TO-15	n-Hexane
GC/MS	EPA TO-15	o-Xylene
GC/MS	EPA TO-15	Styrene
GC/MS	EPA TO-15	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA TO-15	Tetrahydrofuran
GC/MS	EPA TO-15	Toluene
GC/MS	EPA TO-15	trans-1, 2-Dichloroethylene
GC/MS	EPA TO-15	trans-1, 3-Dichloropropylene
GC/MS	EPA TO-15	Trichloroethene (Trichloroethylene)
GC/MS	EPA TO-15	Trichlorofluoromethane (Freon 11)
GC/MS	EPA TO-15	1,1,2-Trichloro1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA TO-15	Vinyl acetate
GC/MS	EPA TO-15	Vinyl chloride
GC/MS	EPA TO-15	Xylenes (Total)

Notes:

1) This laboratory offers commercial testing service.

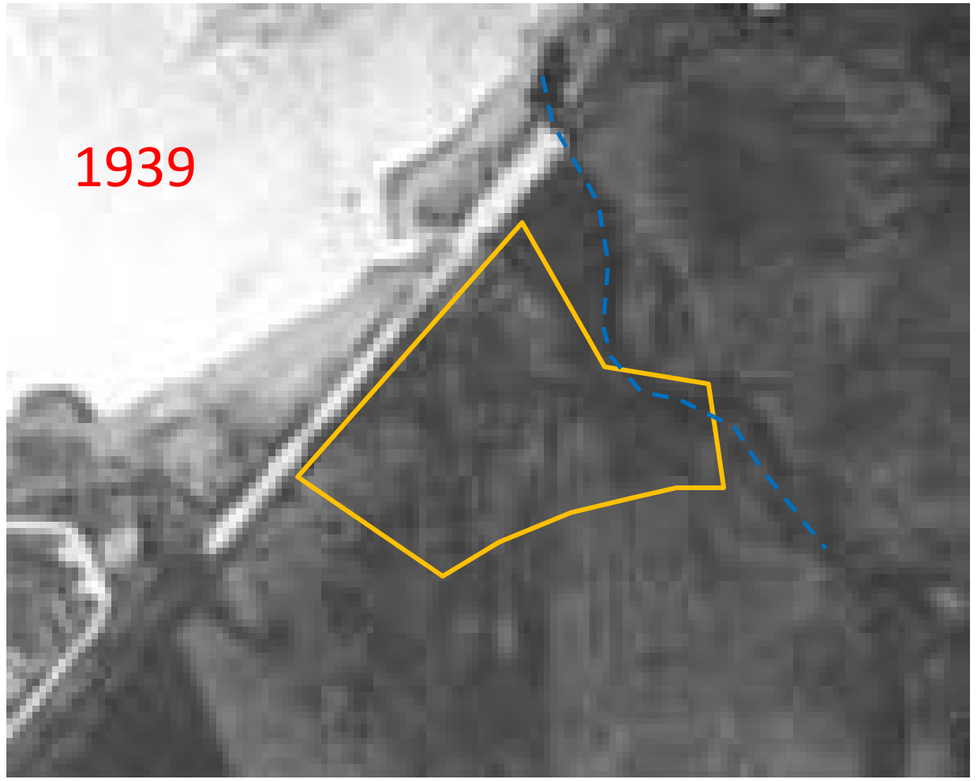
Approved by: _____ Date: February 1, 2013

R. Douglas Leonard
Chief Technical Officer

Re-issued: 2/1/13

Appendix D

Topographic Map and Aerial Photographs

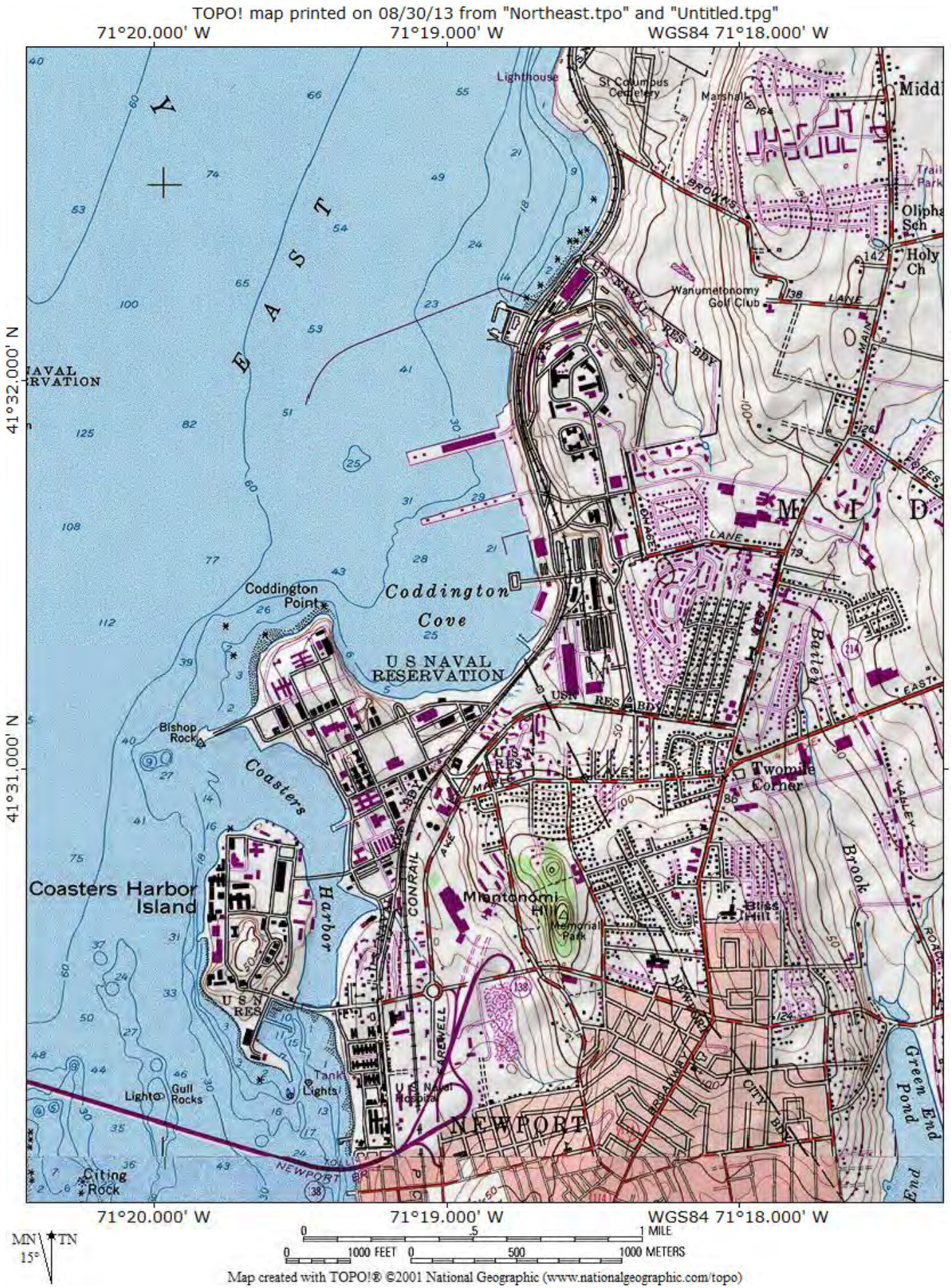

1939

1962

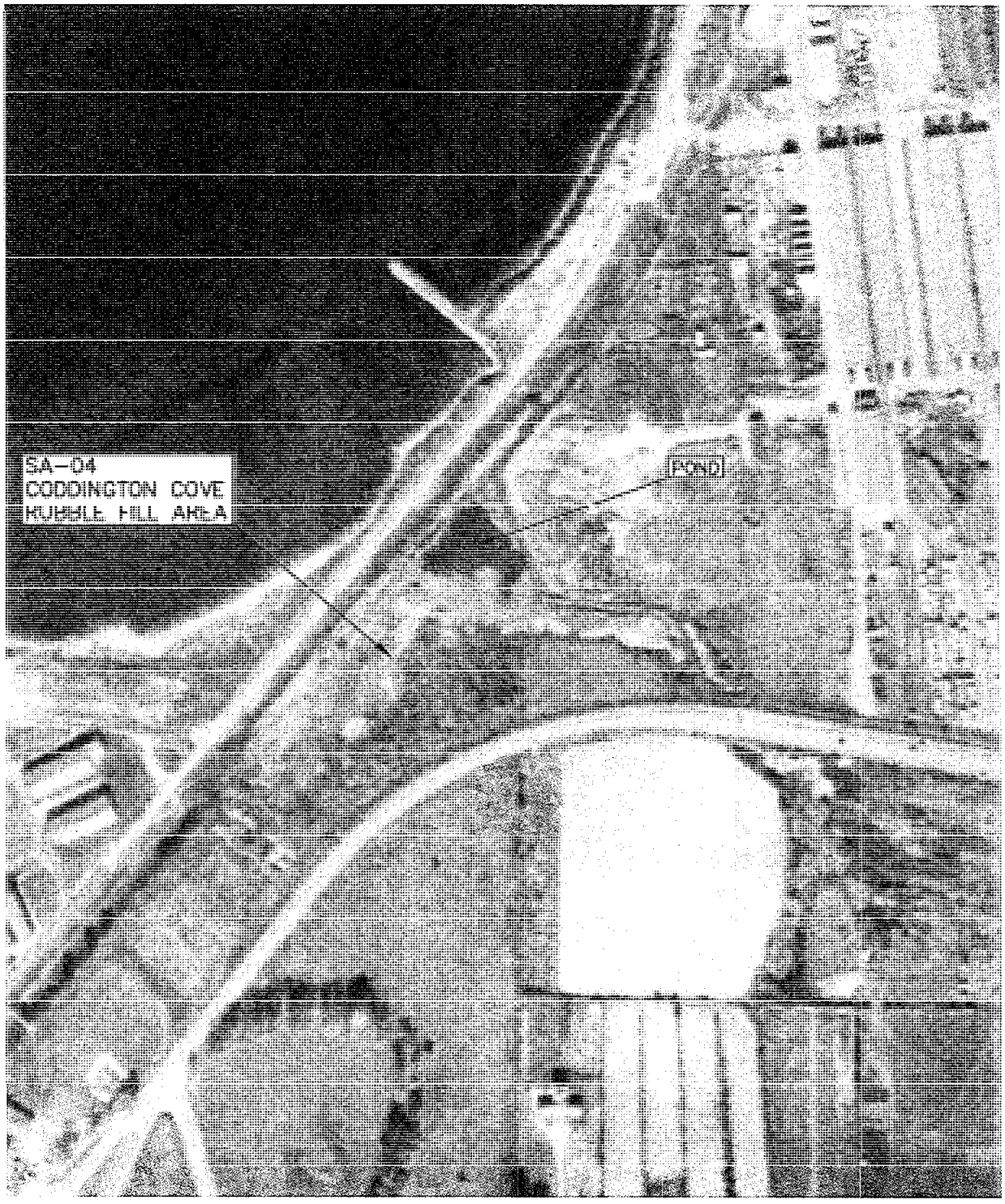
1981

2000s
Railroad Right-of-Way
CCRF Site
Outfall Location
Upgra Area
Auto Repair Shop

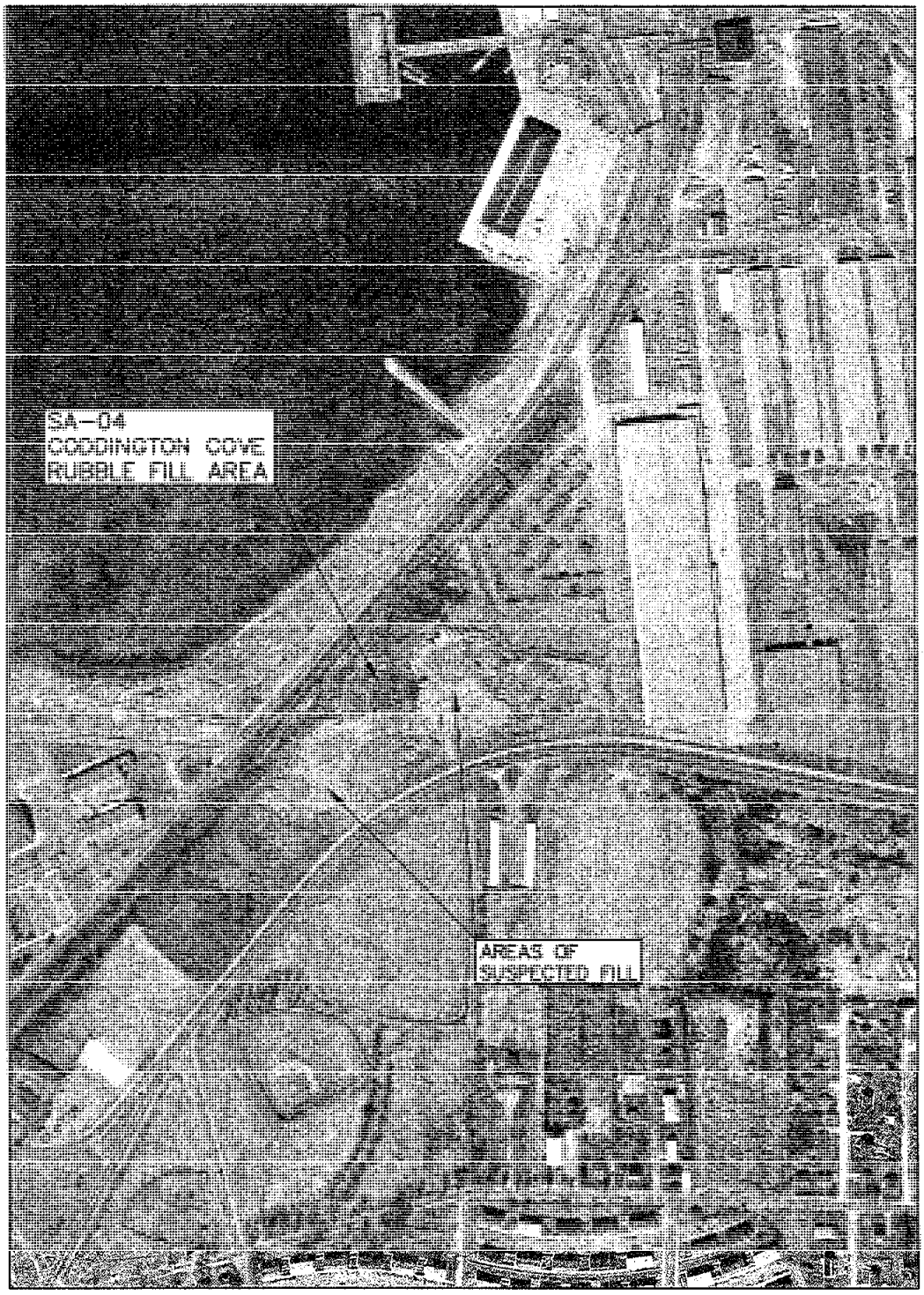
1939



Map created with TOPO!® ©2001 National Geographic (www.nationalgeographic.com/topo)



AERIAL PHOTO – 1951–1952			FIGURE 2–3
CCRF AREA – TECHNICAL MEMORANDUM: RECORD REVIEW AND FIELD SAMPLING PLAN			TETRA TECH NUS, INC.
NAVSTA NEWPORT – NEWPORT, RHODE ISLAND			55 Jonspin Road Wilmington, MA 01887 (978)658-7899
DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	L. SEYDEWITZ	DATE:	MAY 3, 2004
SCALE:	NONE	ACAD NAME:	DWG\5152\0471\FIG_2-3.DWG



AERIAL PHOTO – 1965

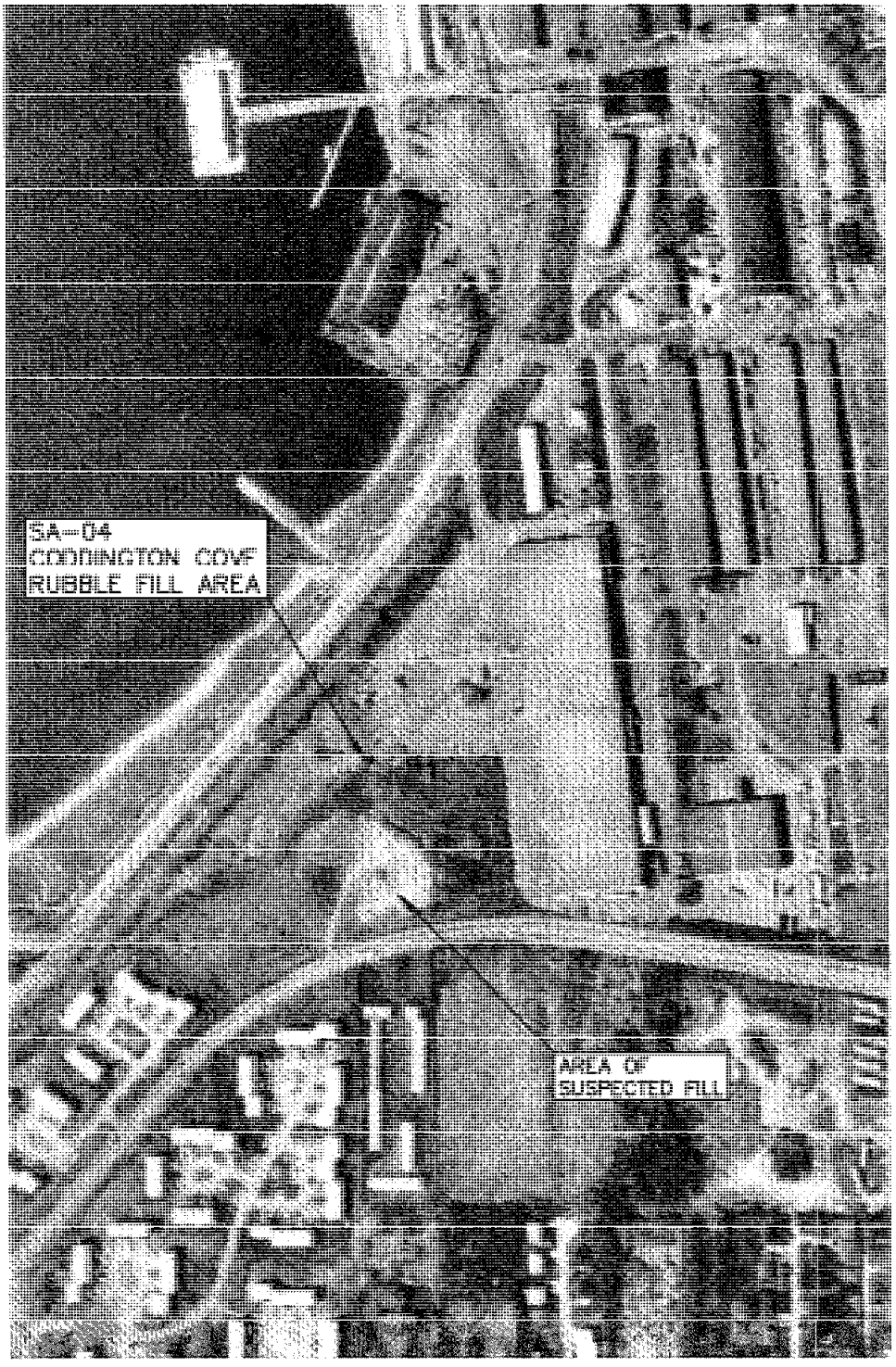
FIGURE 2-4

CCRF AREA – TECHNICAL MEMORANDUM:
RECORD REVIEW AND FIELD SAMPLING PLAN

NAVSTA NEWPORT – NEWPORT, RHODE ISLAND

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	L. SEYDEWITZ	DATE:	MAY 3, 2004
SCALE:	AS NOTED	ACAD NAME:	DWG\5152\0471\FIG_2-4.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658-7899



AERIAL PHOTO – 1981

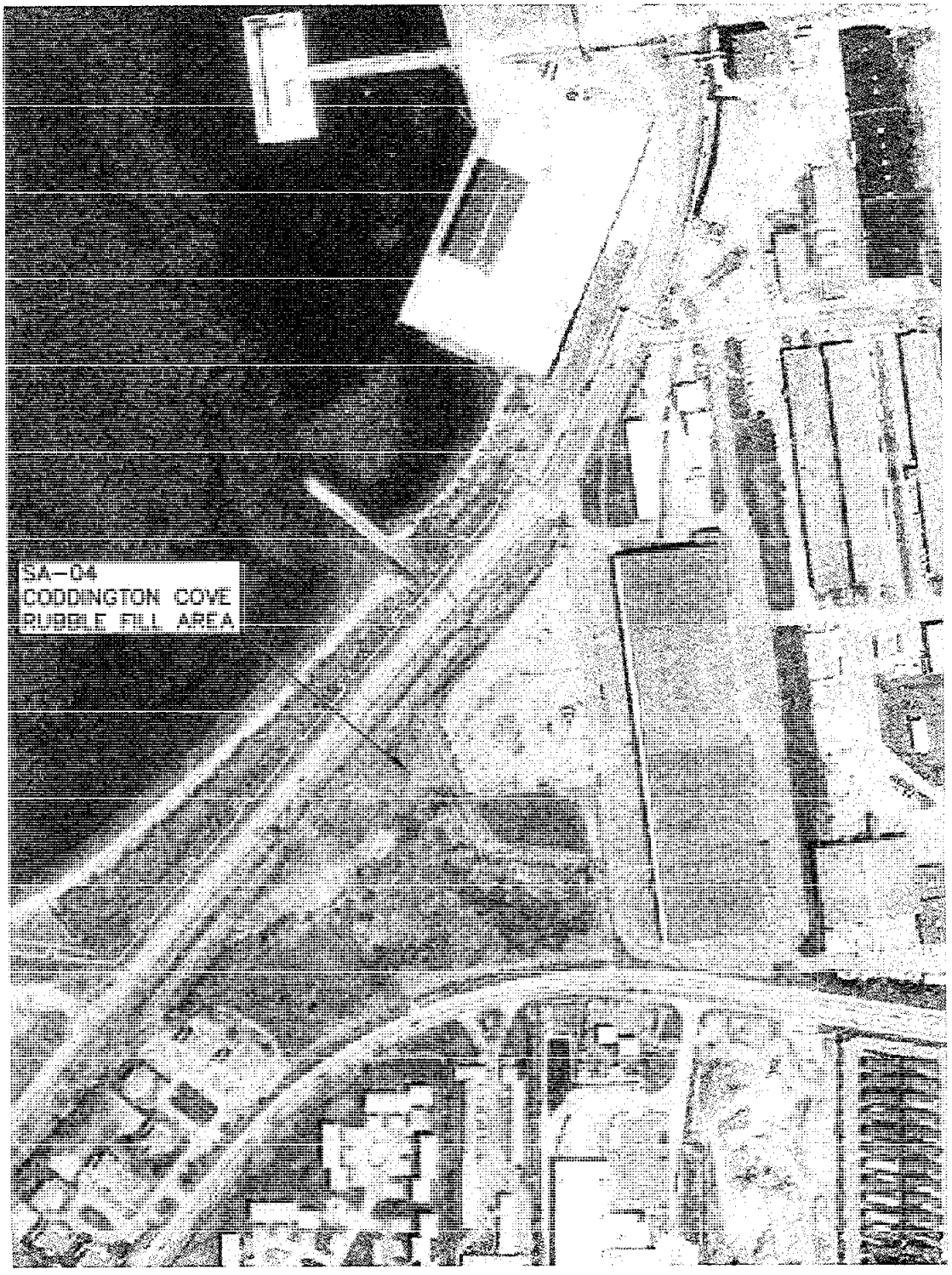
FIGURE 2-5

CCRF AREA – TECHNICAL MEMORANDUM:
RECORD REVIEW AND FIELD SAMPLING PLAN

NAVSTA NEWPORT – NEWPORT, RHODE ISLAND

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	L. SEYDEWITZ	DATE:	MAY 3, 2004
SCALE:	AS NOTED	ACAD NAME:	DWG\5152\0471\FIG_2-5.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658-7899



N

AERIAL PHOTO – 1997

FIGURE 2-6

CCRF AREA – TECHNICAL MEMORANDUM:
RECORD REVIEW AND FIELD SAMPLING PLAN

NAVSTA NEWPORT – NEWPORT, RHODE ISLAND

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	L. SEYDEWITZ	DATE:	MAY 3, 2004
SCALE:	AS NOTED	ACAD NAME:	DWG\5152\0471\FIG_2-6.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658-7899