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SAMPLING AND ANALYSIS PLAN SOURCE AREA INVESTIGATION NIROP FRIDLEY MN
7/1/2013
REOLUTION CONSULTANTS

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SAMPLING AND ANALYSIS PLAN
SOURCE AREA INVESTIGATION
NAVAL INDUSTRIAL RESERVE ORDNANCE PLANT (NIROP)
FRIDLEY, MINNESOTA

Revision: 0

Prepared For:



Department of the Navy
Naval Facilities Engineering Command Midwest
201 Decatur Avenue, Building 1-A
Great Lakes, Illinois, 60088-2801

Prepared By:



Resolution Consultants
A Joint Venture of AECOM & EnSafe
161 Cheshire Lane North, Suite 500
Minneapolis, MN 55441

Contract Number: N62470-11-D-8013
CTO F276

July 2013

SAP WORKSHEET #1: TITLE AND APPROVAL PAGE
(UFP-QAPP Manual Section 2.1)

SAMPLING AND ANALYSIS PLAN

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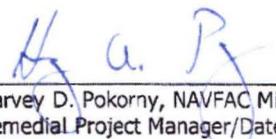
 7/8/13

Cathy Larson, Resolution Consultants
Project Chemist/QA Officer/Date

Electronic Approval in NIRIS doc #9339 on 4/19/2013
Kenneth Bowers, NAVFAC
Quality Assurance Officer/Chemist/Date

 01/08/13

Chris Boehm Carlson, Resolution Consultants
Project Manager/Date

 7/9/13

Harvey D. Pokorny, NAVFAC Midwest
Remedial Project Manager/Date



EXECUTIVE SUMMARY

Resolution Consultants has prepared this Uniform Federal Policy (UFP) Sampling and Analysis Plan (SAP) for a Source Area Investigation at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota, under Contract No. N62470-11-D-8013, Comprehensive Long-term Environmental Action Navy, Contract Task Order F276. This SAP was developed in accordance with applicable Navy, United States (U.S.) Environmental Protection Agency (USEPA) Region 5, and Minnesota Pollution Control Agency (MPCA) requirements, regulations, guidance, and technical standards, as appropriate. This includes the Department of Defense, Department of Energy, and USEPA Interagency Data Quality Task Force environmental requirements regarding federal facilities, as specified in the UFP Quality Assurance Project Plan (UFP-QAPP) guidance (USEPA 2005).

This SAP has been prepared to identify and characterize potential contaminant source areas, affected primarily with trichloroethene (TCE), beneath the NIROP building. For this effort, "source" refers to a mass of TCE-impacted soil or volume of impacted groundwater that persistently acts to degrade soil and/or groundwater quality by releasing dissolved phase TCE (and its degradation byproducts) into the overburden aquifer in such a condition that in-situ remediation could be conducted to accelerate the cleanup time.

This SAP outlines the organization, project management, objectives, planned activities, data acquisition, assessment, oversight, and data review procedures associated with the planned investigation at NIROP. Protocols for sample collection, handling, storage, chain of custody, laboratory and field analyses, data validation, and reporting are also addressed. Field activities conducted under this SAP, which are scheduled to begin in July 2013, will be conducted in accordance with Resolution Consultants' Site-Specific Health and Safety Plan, to be submitted under separate cover.

The purpose of the project is to further define the nature and contaminant concentrations within the subsurface soil and groundwater at the potential source areas and downgradient areas and to collect data needed for designing potential future remedial actions. Based upon results of previous investigations, the presumed source areas appear to be the East Plating Room, which will be a primary focus area for this investigation. Secondary source areas to be investigated are Area of Concern (AOC)-17 in the northwest portion of the building (a former wash rack sump), and the area east and north of monitoring well MS-33I (referred to as 7th and Broadway). The area downgradient of the BAE Systems (BAE) Paint Shop will also be investigated. These potential source areas and the associated downgradient groundwater flow pathways are the focus of this investigation.



The NIROP facility is divided into three separate operable units (OUs) for ease of addressing its contaminant issues:

- Operable Unit 1 (OU1) is the groundwater beneath the entire property,
- Operable Unit 2 (OU2) is the unsaturated soils outside of the building footprint on the NIROP property,
- Operable Unit 3 (OU3) is the unsaturated soils beneath the NIROP building and saturated soil either under or outside the building.

The investigation outlined in this SAP involves groundwater and saturated soil beneath the building and outside the building which is part of OU1 and OU3. This investigation also includes unsaturated soil beneath the building which is part of OU3. No investigation activities are planned for OU2.

Site Background

NIROP is an 83-acre site located about 700 feet east of the Mississippi River in Fridley, Minnesota. Since 1940, the U.S. Navy and its contractors produced advanced weapons systems at the facility. The Navy, USEPA, and MPCA have worked together to complete environmental investigations and remedial efforts at NIROP.

In 1990, USEPA issued a Record of Decision (ROD) for OU1, which included a hydraulic containment remedy for contaminated groundwater using an extraction well system. In September 1992, the groundwater extraction well system came on-line. Through the end of 2011, the system has treated approximately 4.3 billion gallons of groundwater and extracted approximately 71,000 pounds of TCE and other volatile organic compounds.

In 2003, the USEPA issued a ROD for OU2 and OU3 which uses an institutional control (IC), to restrict the property to industrial and limited commercial use until and unless USEPA and MPCA determine that concentrations of hazardous substances in the soil have been reduced to levels which allow for less restrictive uses. For OU2, an IC also prohibits the disturbance or removal of soil deeper than 3 feet below ground surface in two areas north of the building (Area 3A and 4A) without prior written approval of USEPA and MPCA. For OU3, an IC prohibits the disturbance of soil beneath the East Plating Room within the building without prior written approval of the USEPA and MPCA. The IC for OU3 also indicates that the floor of the East Plating Room (concrete pit floor 8 to 12 feet below grade floor) is an Engineering Control and will not be removed without prior written approval of USEPA and MPCA.



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In 1997, an investigation of OU3, as part of the Remedial Investigation (RI) study to locate source areas beneath the building, included analysis of approximately 500 soil samples to ascertain areas of maximum contamination. Results of this effort suggest three potential source areas:

1. East Plating Room;
2. AOC-17 (a former wash rack sump); and
3. Area east and north of monitoring well MS-33I (7th and Broadway).

Based upon more recent data collected after the RI, it appears that dispersion from a BAE Resource Conservation and Recovery Act Paint Shop source area (south of Navy-controlled property) may have affected groundwater beneath OU3.

Elevated concentrations of TCE and its degradation products in these potential source areas may contribute to elevated concentrations of TCE and its degradation products in the groundwater beneath the NIROP facility and thereby force the groundwater extraction and treatment system to operate indefinitely. Although the groundwater treatment system is operating in accordance with the ROD, it may take a very long time to reach cleanup goals if the potential source areas continue to provide elevated concentrations of TCE to the groundwater.

The Navy is continuously monitoring the system and evaluating new cleanup options to identify actions that may help speed up the process and allow the site to reach cleanup goals faster. To this end, the Navy has elected to undertake this source area investigation to better define the nature and contaminant concentrations within the subsurface soil and groundwater at the potential source areas. This will be accomplished using qualitative screening with a membrane interface probe along with quantitative soil and groundwater sampling with laboratory analysis. Explorations will extend from beneath the floor to a depth of approximately 80 feet. The goals are to develop data necessary to identify potential source areas and to assess potential source area control alternatives.



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FIGURE

Figure 10-1 Site Layout and Proposed Boring Locations

APPENDICES

- Appendix A Reference Figures from Previous Reports
- Appendix B Resolution Consultants Standard Operating Procedures, Field Forms, and Data Validation Checklists
- Appendix C Laboratory Control Limits, Standard Operating Procedures and Laboratory Certifications
- Appendix D Subcontractor Standard Operating Procedures



ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
%D	percent difference or percent drift
%RSD	relative standard deviation
AOC	Area of Concern
BAE	BAE Systems
bgs	below ground surface
CERCLA	Comprehensive Environmental Response, Compensation & Liability Act
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-term Environmental Action Navy
COC	chain of custody
CSM	conceptual site model
CTO	Contract Task Order
DO	dissolved oxygen
DoD	Department of Defense
DVM	Data Validation Manager
EC	electrical conductivity
ECD	electron capture detector
ELAP	Environmental Laboratory Accreditation Program
EZVI	emulsified zero valent iron
FID	flame ionization detector
FOL	Field Operations Leader
GC/MS	gas chromatograph/mass spectrometer
IC	institutional control
ICAL	initial calibration
ICP	inductively coupled plasma spectroscopy
ICV	initial calibration verification
IDW	investigation-derived waste
LCS/LCSD	laboratory control sample/laboratory control sample duplicate
LOD	limit of detection
LOQ	limit of quantitation
MCL	maximum contaminant level
MDH	Minnesota Department of Health
mg/kg	milligram per kilogram
MIP	membrane interface probe
MPCA	Minnesota Pollution Control Agency
MS/MSD	matrix spike/matrix spike duplicate
NA	not applicable
NAPL	nonaqueous phase liquid
NAVFAC	Naval Facilities Engineering Command
NAVFAC LANT	Naval Facilities Engineering Command, Atlantic
NAVFAC MW	Naval Facilities Engineering Command, Midwest
NIRIS	Naval Installation Restoration Information Solution
NIROP	Naval Industrial Reserve Ordnance Plant



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ORP	oxidation-reduction potential
OU	Operable Unit
PAL	project action limit
PC	Project Chemist
PCB	polychlorinated biphenyl
PID	photoionization detector
PM	Project Manager
ppm	part per million
QA	quality assurance
QAO	Quality Assurance Officer
QC	quality control
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
ROD	Record of Decision
RPD	relative percent difference
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SRV	Soil Reference Value
SSO	Site Safety Officer
SVOCs	semivolatile organic compounds
TCE	trichloroethene
TCLP	toxicity characteristic leaching procedure
Tetra Tech	Tetra Tech NUS, Inc.
TOC	total organic carbon
TriMatrix	TriMatrix Laboratories
UFP	Uniform Federal Policy
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
µg/L	microgram per liter
U.S.	United States
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound
XSD	halogen specific detector



SAP WORKSHEET #2: SAMPLING AND ANALYSIS PLAN IDENTIFYING INFORMATION

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota
 Operable Unit (OU) 1 and OU3

Contractor Name: Resolution Consultants

Contract Number: N62470-11-D-8013

Contract Title: Comprehensive Long-term Environmental Action Navy (CLEAN)

Work Assignment Number: Contract Task Order (CTO) F276

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of:
 - The Intergovernmental Data Quality Task Force *Uniform Federal Policy for Quality Assurance (QA) Plans (UFP-QAPP)* (United States [U.S.] Environmental Protection Agency [USEPA] 2005).
 - *Guidance for QA Project Plans*, USEPA QA/G-5 (USEPA 2002).
2. Identify regulatory program(s):
 - Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980, as reauthorized by Superfund Amendments and Reauthorization Act.
 - Minnesota Environmental Response Liability Act of 1983.
3. This document is a project-specific SAP.
4. List dates of scoping sessions that were held:

Scoping Session	Date
Internal Naval Facilities Engineering Command (NAVFAC) Scoping Session	May 30, 2012
Internal NAVFAC Scoping Session	July 15, 2012
Internal NAVFAC Scoping Session	September 3, 2012
NIROP Team Meeting	October 9-10, 2012



Scoping Session	Date
Internal NAVFAC Scoping Session	January 25, 2013

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

Title	Date
Remedial Action Work Plan, Tetra Tech NUS, Inc. (Tetra Tech) Filed in the NIROP Facility Administrative Record as N91192_000750	September 2005

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

Regulatory Oversight — USEPA, Region 5

Regulatory Oversight — Minnesota Pollution Control Agency (MPCA)

7. Lead organization:

Department of the Navy, Naval Facilities Engineering Command, Midwest (NAVFAC MW)

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:

All requirements are included in this SAP.

9. A cross-reference for this SAP is presented in the following table.

Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
<i>Documentation</i>		
1	Title and Approval Page	1-1
2	Sampling and Analysis Plan Identifying Information	2-1
3	Distribution List	3-1
4	Project Personnel Sign-Off Sheet	4-1
<i>Project Organization</i>		
5	Project Organizational Chart	5-1
6	Communication Pathways	6-1



Worksheet #	Required Information	Crosswalk to Related Information
7	Personnel Responsibilities Table	7-1
8	Special Personnel Training Requirements Table	8-1
<i>Project Planning/ Problem Definition</i>		
9	Project Scoping Session Participants Sheet	9-1
10	Problem Definition	10-1
11	Project Quality Objectives/Systematic Planning Process Statements	11-1
12	Field Quality Control Samples	12-1
13	Sources of Secondary Data Criteria and Limitations Table	13-1
14	Summary of Project Tasks	14-1
15	Reference Limits and Evaluation Tables	15-1
16	Project/Timeline Table	16-1
B. Measurement Data Acquisition		
<i>Sampling Tasks</i>		
17	Sampling Design and Rationale	17-1
18	Location-Specific Sampling Methods/SOP Requirements Table	18-1
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20	Field Quality Control Sample Summary Table	20-1
21	Project Sampling SOP References Table	21-1
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	22-1
<i>Analytical Tasks</i>		
23	Analytical SOP References Table	23-1
24	Analytical Instrument Calibration Table	24-1
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	25-1
<i>Sample Collection</i>		
26	Sample Handling System	26-1
27	Sample Custody Requirements	27-1
<i>Quality Control Samples</i>		
28	Laboratory QC Samples Table	28-1
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29	Project Documents and Records Table	29-1
30	Analytical Services Table	30-1
C. Assessment Oversight		
31	Planned Project Assessments Table	31-1
32	Assessment Findings and Corrective Action Responses Table	32-1
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D. Data Review		
34-36	Data Verification and Validation (Steps I and II/IIB) Process Table	34-36-1
37	Usability Assessment	37-1



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*Sampling and Analysis Plan — Source Area Investigation
Naval Industrial Reserve Ordnance Plant, Fridley Minnesota
SAP Worksheet #2
Revision: 0; July 2013*

Notes:

QC quality control
SOP standard operating procedure



SAP WORKSHEET #3: DISTRIBUTION LIST

(UFP-QAPP Manual Section 2.3.2)

Distribution List				
SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Harvey Pokorny	RPM	Department of the Navy	(847) 688-2600 x611	harvey.pokorny@navy.mil
Val Jurka	Environmental Engineer	Department of the Navy	(757) 322-8319	val.jurka@navy.mil
Howard Hickey	Restoration PM	Department of the Navy	(847) 688-2600 x243	howard.hickey@navy.mil
Teresè Van Donsel	Environmental Engineer	Department of the Navy	(847) 688-2600	terese.vandonsel@navy.mil
Sheila Desai	RPM	USEPA Region 5	(312) 353-4150	desai.sheila@epa.gov
Deepa de Alwis	PM	MPCA	(651) 757-2572	deepa.dealwis@state.mn.us
Chris Boehm Carlson	CTO PM	Resolution Consultants	(763) 551-2439	chris.boehm@aecom.com
Dan Phelps	Field Operations Leader and Site Safety Officer	Resolution Consultants	(651) 367-2305	daniel.c.phelps@aecom.com
James Buss	Project Hydrogeologist	Resolution Consultants	(608) 828-8210	james.buss@aecom.com
Scott Tarmann	Project Engineer	Resolution Consultants	(414) 944-6184	scott.tarmann@aecom.com
Cathy Larson	PC /QAO	Resolution Consultants	(763) 551-2474	cathy.larson@aecom.com
Stephanie Warino	CTO PM (outgoing)	Tetra Tech	(412) 921-8868	stephanie.Warino@tetrattech.com
Paul Walz	PM	Bay West	(651) 291-3491	paulw@baywest.com
Walt Roudebush	Laboratory PC	TriMatrix Laboratories, Inc.	(616) 975-4561	roudebushw@trimatrixlabs.com

Notes:

Each person listed in this table will be responsible for distributing copies of this SAP to appropriate personnel within their organization.

- CTO = Contract Task Order
- MPPA = Minnesota Pollution Control Agency
- PC = Project Chemist
- PM = Project Manager
- RPM = Remedial Project Manager
- QAO = Quality Assurance Officer
- SAP = Sampling and Analysis Plan
- Tetra Tech = Tetra Tech NUS, Inc.
- USEPA = United States Environmental Protection Agency



SAP WORKSHEET #4: PROJECT PERSONNEL SIGN-OFF SHEET

(UFP-QAPP Manual Section 2.3.2)

Certification that project personnel have read the text will be obtained by one of the following methods as applicable:

1. In the case of regulatory agency personnel with oversight authority, approval letters or e-mails will constitute verification that applicable sections of the SAP have been reviewed. Copies of regulatory agency approval letters/e-mails will be retained in the project files and are listed in Worksheet #29 as project records.

2. E-mails will be sent to the Navy, Resolution Consultants, and subcontractor project personnel who will be requested to verify by e-mail that they have read the applicable SAP/sections and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files and is identified in Worksheet #29.

A copy of the signed Worksheet #4 will be retained in the project files and is identified as a project document in Worksheet #29.



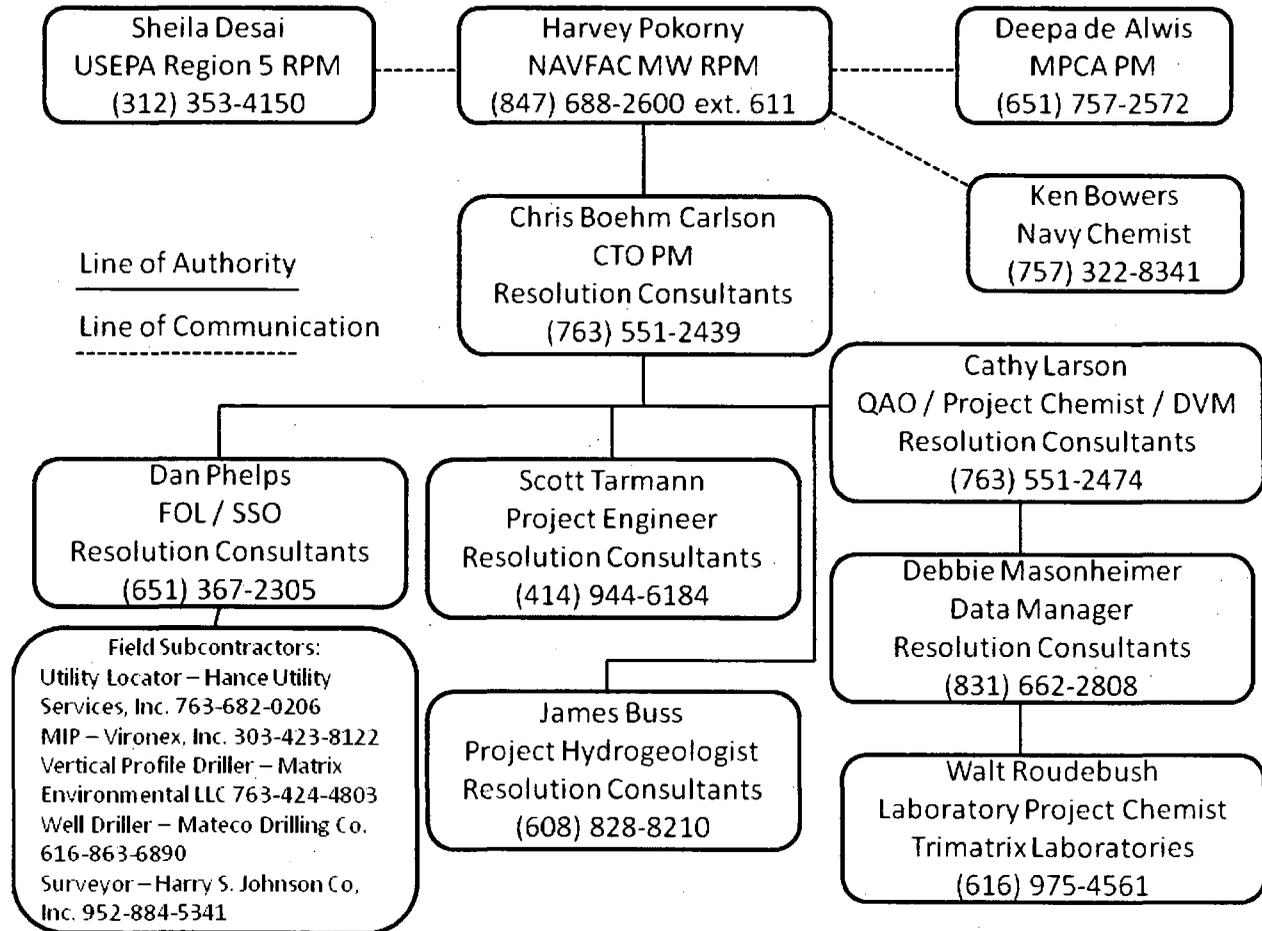
Project Personnel Sign-Off Sheet					
Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulator Partnering Team Personnel					
Harvey Pokorny	Navy RPM (Manages project for the Navy)	(847) 688-2600 x611		All	
Sheila Desai	USEPA RPM (Provides Regulatory Oversight)	(312) 353-4150		All	
Deepa de Alwis	MPCA PM (Provides Regulatory Oversight)	(651) 757-2572		All	
Resolution Consultants' Partnering Team Personnel					
Chris Boehm Carlson	CTO PM (Manages project for Resolution Consultants)	(763) 551-2439		All	
Dan Phelps	Field Operations Leader and Site Safety Officer (Oversees field activities and site safety)	(651) 367-2305		All	
Cathy Larson	PC/QAO (Oversees quality and subcontracted analytical services)	(763) 551-2474		All	
Scott Tarmann	Project Engineer (Oversees engineering services)	(414) 944-6184		All	
James Buss	Project Hydrogeologist (Provides senior level hydrogeological services)	(608) 828-8210		All	
Subcontractor Personnel					
Walt Roudebush	TriMatrix Laboratories/Laboratory PC (Representative for subcontracted analytical services)	(616) 975-4561		Worksheets #6, #12, #14, #15, #19, #20, #23-28, #30, and #34-36	

Notes:

- CTO = Contract Task Order
- MCPA = Minnesota Pollution Control Agency
- PC = Project Chemist
- PM = Project Manager
- RPM = Remedial Project Manager
- QAO = Quality Assurance Officer
- SAP = Sampling and Analysis Plan
- USEPA = United States Environmental Protection Agency

SAP WORKSHEET #5: PROJECT ORGANIZATIONAL CHART

(UFP-QAPP Manual Section 2.4.1)



Notes:

CTO = Contract Task Order
 DVM = Data Validation Manager
 FOL = Field Operations Leader
 MIP = Membrane Interface Probe
 MPCA = Minnesota Pollution Control Agency
 NAVFAC MW = Naval Facilities Engineering Command Midwest
 PM = Project Manager

QAO = Quality Assurance Officer
 RPM = Remedial Project Manager
 SSO = Site Safety Officer
 USEPA = United States Environmental Protection Agency



SAP WORKSHEET #6: COMMUNICATION PATHWAYS

(UFP-QAPP Manual Section 2.4.2)

Communication pathways for the SAP are shown below.

Communication Pathways				
Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Regulatory Agency Interface	USEPA RPM	Sheila Desai	(312) 353-4150	The NAVFAC MW RPM will contact each regulatory agency via phone and/or e-mail within 48 hours when significant corrective actions or changes to the SAP occur. This includes notification of significant analytical data quality issues or equipment failure.
	MPCA PM	Deepa de Alwis	(651) 757-2572	
	NAVFAC MW RPM	Harvey Pokorny	(847) 688-2600 x611	
Field Progress Reports	Resolution Consultants PM	Chris Boehm Carlson	(763) 551-2439	The Resolution Consultants FOL will contact the Resolution Consultants PM on a daily basis via phone, and every 1-2 days summarizing progress via e-mail.
	Resolution Consultants FOL	Dan Phelps	(651) 367-2305	
Gaining Site Access	NAVFAC MW RPM	Harvey Pokorny	(847) 688-2600 x611	The Resolution Consultants FOL will contact the NAVFAC MW RPM, or designee, verbally or via e-mail at least 30 days prior to commencement of field work to arrange for access to the site for all field personnel. Notification will be provided to facility owner and BAE.
	Resolution Consultants FOL	Dan Phelps	(651) 367-2305	
	Cassidy Turley (Property Managers)	Tom Vierling	(612) 341-4444	
Obtaining Utility Clearances for Intrusive Activities	Resolution Consultants FOL	Dan Phelps	(651) 367-2305	A minimum of two weeks prior to the commencement of any intrusive activities, Resolution Consultants will coordinate utility clearance with the Navy and the Gopher State One Call public utility locating service. A private utility locator may also be contracted to locate private utilities at the site. The Resolution Consultants FOL will document the utility clearance process.
	NAVFAC MW RPM	Harvey Pokorny	(847) 688-2600 x611	
Stop Work Due to Safety Issues	Resolution Consultants PM	Chris Boehm Carlson	(763) 551-2439	If Resolution Consultants is the responsible party for a stop work command, then the Resolution Consultants SSO will inform on-site personnel, subcontractor(s), and the Resolution Consultants PM within 1 hour (verbally or by e-mail).
	Resolution Consultants FOL/SSO	Dan Phelps	(651) 367-2305	
	NAVFAC MW RPM	Harvey Pokorny	(847) 688-2600 x611	The Resolution Consultants PM will notify the NAVFAC MW RPM within 1 hour of notification (verbally or by e-mail). If a subcontractor is the responsible party for a stop work command, then the subcontractor PM must verbally inform the Resolution Consultants SSO within 15 minutes, and the Resolution Consultants SSO will then follow the procedure listed above.
	Cassidy Turley (Property Managers)	Tom Vierling	(612) 341-4444	
	BAE	Tim Ruda	763-572-6906	



Communication Pathways				
Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
SAP Changes Prior to Field/ Laboratory Work	Resolution Consultants FOL	Dan Phelps	(651) 367-2305	<p>The Resolution Consultants PM will document the proposed changes via a Field Task Modification Request form within 5 days and send the NAVFAC MW RPM a concurrence letter within 7 days of identifying the need for change, if necessary. The RPM can authorize submittal of a concurrence letter and can request changes to funding. The AQ is the only entity that can formally commit funds.</p> <p>SAP amendments will be submitted by the Resolution Consultants PM to the NAVFAC MW RPM for review and approval.</p> <p>The Resolution Consultants PM will send scope changes to the Partnering Team via e-mail within 1 business day.</p>
	Resolution Consultants PM	Chris Boehm Carlson	(763) 551-2439	
	NAVFAC MW RPM	Harvey Pokorny	(847) 688-2600 x611	
SAP Changes in the Field	Resolution Consultants FOL	Dan Phelps	(651) 367-2305	<p>The Resolution Consultants FOL will verbally inform the Resolution Consultants PM on the day that the issue is discovered.</p> <p>The Resolution Consultants PM will inform the NAVFAC MW RPM (verbally or via e-mail) within 1 business day of discovery.</p> <p>The NAVFAC MW RPM will issue a scope change (verbally or via e-mail), if warranted. The scope change is to be implemented before further work is executed.</p> <p>The Resolution Consultants PM will document the change via a Field Task Modification Request form within 2 days of identifying the need for change and will obtain required approvals within 5 days of initiating the form.</p>
	Resolution Consultants PM	Chris Boehm Carlson	(763) 551-2439	
	NAVFAC MW RPM	Harvey Pokorny	(847) 688-2600 x611	
Field Corrective Actions	Resolution Consultants PM	Chris Boehm Carlson	(763) 551-2439	<p>The Resolution Consultants FOL will notify the Resolution Consultants PM verbally or by e-mail within one business day that the corrective action has been completed.</p> <p>The Resolution Consultants PM will then notify the NAVFAC MW RPM (verbally or by e-mail) within 1 business day.</p>
	Resolution Consultants FOL	Dan Phelps	(651) 367-2305	
	NAVFAC MW RPM	Harvey Pokorny	(847) 688-2600 x611	



Communication Pathways				
Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Sample Receipt Variances	Resolution Consultants PM	Chris Boehm Carlson	(763) 551-2439	<p>The Laboratory PC will notify (verbally or via e-mail) the Resolution Consultants PC immediately upon receipt of any chain-of-custody/sample receipt variances for clarification or direction.</p> <p>The Resolution Consultants PC will notify (verbally or via e-mail) the Resolution Consultants PM within 1 business day, if corrective action is required.</p> <p>The Resolution Consultants PM will notify (verbally or via e-mail) the Resolution Consultants FOL within 1 business day of any required corrective action.</p> <p>The Resolution Consultants PC will notify (verbally or via e-mail) the laboratory PC within 1 business day, if corrective action is required.</p>
	Resolution Consultants PC	Cathy Larson	(763) 551-2474	
	TriMatrix Laboratory PC	Walt Roudebush	(616) 975-4561	
Analytical Corrective Actions	TriMatrix Laboratory PC	Walt Roudebush	(616) 975-4561	<p>The laboratory shall notify the Resolution Consultants PC of any analytical data anomaly within 1 business day of discovery. After the laboratory receives guidance from Resolution Consultants' PC, the laboratory shall initiate any corrective action to prevent further anomalies.</p>
	Resolution Consultants PC	Cathy Larson	(763) 551-2474	
Analytical Data Quality Issues	TriMatrix Laboratory PC	Walt Roudebush	(616) 975-4561	<p>The laboratory PC notifies (verbally or via e-mail) the Resolution Consultants' PC within 1 business day of when an issue related to laboratory data is discovered. Resolution Consultants' PC notifies Resolution Consultants' PM within 1 business day.</p> <p>Resolution Consultants PC notifies the Resolution Consultants' PM verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has been detected that could affect this project and/or other projects. Resolution Consultants PM verbally advises the Navy RPM within 24 hours of notification from the PC. The Navy RPM will engage the NAVFAC LANT chemist to ensure the issues with this project can be evaluated to determine impact to other DoD projects.</p>
	Resolution Consultants PC	Cathy Larson	(763) 551-2474	
	Resolution Consultants PM	Chris Boehm Carlson	(763) 551-2439	
	NAVFAC MW RPM	Harvey Pokorny	(847) 688-2600 x611	



Communication Pathways				
Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Reporting Laboratory Quality Variances	Resolution Consultants PC/DVM	Cathy Larson	(763) 551-2474	<p>The laboratory PC will notify (verbally or via e-mail) the Resolution Consultants PC of any variance from the quality limits identified in this SAP on the day that the variance becomes known.</p> <p>The Resolution Consultants PC /DVM will notify (verbally or via e-mail) the Resolution Consultants PM within 1 business day of the need for corrective action, if the variance is a significant issue.</p> <p>The Resolution Consultants PM will notify (verbally or via e-mail) the NAVFAC MW RPM within 1 business day.</p> <p>The Laboratory PC will document all quality variances in the Case Narrative of the Analytical Laboratory Report.</p>
	Resolution Consultants PM	Chris Boehm Carlson	(763) 551-2439	
	Resolution Consultants FOL	Dan Phelps	(651) 367-2305	
	TriMatrix Laboratory PC	Walt Roudebush	(616) 975-4561	
Notification of Non-Usable Data	TriMatrix Laboratory PC	Walt Roudebush	(616) 975-4561	<p>If the laboratory determines that any data they have generated is non-usable, the laboratory PC will notify (verbally or via e-mail) the Resolution Consultants PC within 1 business day of when the issue is discovered.</p> <p>The Resolution Consultants PC will notify (verbally or via e-mail) the Resolution Consultants DVM and the Resolution Consultants PM within 1 business day of the need for corrective action, if the non-usable data is a significant issue (i.e., critical sample data). Corrective action may include resampling and/or reanalyzing the effected samples.</p> <p>If a Resolution Consultants Data Validator identifies non-usable data during the data validation process, the Resolution Consultants DVM will notify the Resolution Consultants PM verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has resulted in non-usable data.</p> <p>The Resolution Consultants PM will take corrective action appropriate for the identified deficiency to ensure the project objectives are met.</p> <p>The Resolution Consultants PM will notify (verbally or via email) the NAVFAC MW RPM on any problems with the laboratory or analysis that could significantly affect the usability of the data or project failures that impact the ability to complete the scope of work. Such notification will be made within 1 business day of when the issue is discovered. The Navy RPM will engage the NAVFAC LANT chemist to ensure issues with this project can be evaluated to determine impact to other DoD projects.</p>
	Resolution Consultants PC/DVM	Cathy Larson	(763) 551-2474	
	Resolution Consultants PM	Chris Boehm Carlson	(763) 551-2439	
	NAVFAC MW RPM	Harvey Pokorny	(847) 688-2600 x611	



**RESOLUTION
CONSULTANTS**

*Sampling and Analysis Plan — Source Area Investigation
Naval Industrial Reserve Ordnance Plant, Fridley Minnesota
SAP Worksheet #6
Revision: 0; July 2013*

Notes:

AQ = Acquisition
BAE = BAE Systems
DoD = Department of Defense
USEPA = United States environmental Protection Agency
MPCA = Minnesota Pollution Control Agency
NAVFAC LANT = Department of the Navy, Naval Facilities Engineering Command, Atlantic
NAVFAC MW = Department of the Navy, Naval Facilities Engineering Command, Midwest
FOL = Field Operations Leader
RPM = Remedial Project Manager
SAP = Sampling and Analysis Plan
SSO = Site Safety Office
PC = Project Chemist
PM = Project Manger
DVM = Data Validation Manager



SAP WORKSHEET #7: PERSONNEL RESPONSIBILITIES TABLE

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Howard Hickey	NAVFAC Program Manager	NAVFAC	Oversees resourcing and participates in evaluation of scope modifications and overall technical goals.
Harvey Pokorny	NAVFAC RPM/Manages project activities for the Navy	NAVFAC MW	Primary Point of Contact for the Navy. Oversees project implementation, including scoping, data review, and evaluation, on behalf of the Navy. Will distribute a signed copy of the SAP to all team members. Authority to stop work. Notifies regulators of problems that require corrective action, corrective actions that are initiated, and schedule changes.
Sheila Desai	RPM/Regulatory Support	USEPA Region 5	Makes sure that investigation work and documentation are in compliance with applicable federal regulations. Represents the interests of USEPA with regard to project expectations and requirements of existing decision documents.
Deepa de Alwis	MPCA PM	MPCA	Makes sure that investigation work and documentation are in compliance with applicable Minnesota regulations. Represents the interests of MPCA with regard to project expectations and requirements of existing decision documents.
Terese Van Donsel	Navy QA Project Officer	NAVFAC MW	Participates in evaluation of potential scope modifications and advises on CERCLA process, data utilization, and technical approach.
Ken Bowers	QAM	NAVFAC LANT	Reviews SAP to ensure that the SAP meets Navy requirements. SAP approval.
Ken Brown	Regional Program Manager/Manages CLEAN program in Midwest	Resolution Consultants	Oversees CLEAN program on behalf of Resolution Consultants in the Midwest.
John Knopf	Health and Safety Manager	Resolution Consultants	Oversees CLEAN program Health and Safety Program.
Chris Boehm Carlson	Contractor PM for CTO /Manages project on a daily basis	Resolution Consultants	Oversees project, financial, scheduling and technical day-to-day management of the contracted scope of work. Overall coordination of the project and document review. Will distribute a signed copy of the SAP to all Resolution Consultant team members.



Name	Title/Role	Organizational Affiliation	Responsibilities
Dan Phelps	Field Operations Leader/ Site Safety Officer	Resolution Consultants	<p>Supervises, coordinates, and performs field activities. Ensures that all health and safety requirements unique to this project are implemented.</p> <p>Functions as the on-site communication link between field staff members, NAVAC MW, and the Resolution PM.</p> <p>Ensures the proper maintenance of site logbooks, field logbooks, and field recordkeeping.</p> <p>Identifies and resolves problems in the field, resolving difficulties via consultation with Resolution Consultants PM and NAVFAC MW RPM, implements and documents corrective action procedures, and provides communication between the field team and project management.</p> <p>Responsible for site safety and maintenance of project-specific safety-related documentation.</p> <p>Ensures that field personnel comply with all procedures established in the Health and Safety Plan.</p> <p>Ensures that facility personnel and subcontractors are adequately advised and kept clear of potentially contaminated materials.</p> <p>Terminates work if an imminent safety hazard, emergency situation, or other potentially dangerous situation is encountered.</p>
Cathy Larson	QAO/Oversees project QA activities	Resolution Consultants	Assist in SAP preparation. Makes certain quality aspects of the CLEAN program are implemented.
Cathy Larson	PC/Data Validation Manager	Resolution Consultants	Prepares laboratory scope, coordinates with laboratory, and performs data quality review. Coordinates analyses with laboratory chemists, makes sure the scope is followed, reviews QA data packages and communicates with Resolution Consultants staff. Manages data validation activities.
Scott Tarmann	Project Engineer	Resolution Consultants	Oversee engineering services, including providing input on scope of investigation and data requirements for remedial design.
Jim Buss	Project Hydrogeologist	Resolution Consultants	Provide senior level hydrogeological services, including input in report preparation and drilling subcontractor procurement.
Debbie Masonheimer	Data Manager	Resolution Consultants	Assists in SAP preparation, makes sure that data management activities are performed in accordance with SAP procedure.



Name	Title/Role	Organizational Affiliation	Responsibilities
Walt Roudebush	Laboratory PC/Analytical Subcontractor	TriMatrix Laboratories	Primary laboratory contact; ensures analyses are performed in accordance with SAP protocols.

Notes:

CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act
CLEAN = Comprehensive Long-term Environmental Action Navy
CTO = Contract Task Order
MPCA = Minnesota Pollution Control Agency
NAVFAC = Naval Facilities Engineering Command
NAVFAC LANT = Naval Facilities Engineering Command, Atlantic
NAVFAC MW = Naval Facilities Engineering Command, Midwest
PC = Project Chemist
PM = Project Manager
QA = Quality Assurance
QAM = Quality Assurance Manager
QAO = Quality Assurance Officer
RPM = Remedial Project Manager
SAP = Sampling and Analysis Plan
USEPA = United States Environmental Protection Agency



SAP WORKSHEET #8: SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

(UFP-QAPP Manual Section 2.4.4)

Field personnel will follow the field Standard Operating Procedures (SOPs) in Appendix B. Laboratory staff will follow the analytical SOPs in Appendix C. Laboratory certifications are also included in Appendix C.

There are no other special personnel training required for the execution of field activities under this CTO.



SAP WORKSHEET #9: PROJECT SCOPING SESSION PARTICIPANTS SHEET

(UFP-QAPP Manual Section 2.5.1)

Project Name: Source Area Groundwater Investigation			Site Name: Naval Industrial Reserve Ordnance Plant (NIROP)		
Projected Date(s) of Sampling: Summer 2013			Site Location: Fridley, Minnesota		
Project Manager: Chris Boehm Carlson					
Date of Sessions: May 30, 20/12, July 15, 2012, September 3, 2012, and January 25, 2013					
Participants: Internal NAVFAC Scoping Sessions					
Scoping Session Purpose: Developed objectives and scope of the project					
Comments/Decisions: Sessions resulted in information presented in the "Contract N62470-11-D-8013, CTO F276: CLEAN at the Naval Industrial Reserve Ordnance Plant Fridley, Minnesota" and the "NIROP Fridley UFP-SAP/QAPP for Source Area Groundwater Investigation, Draft", March 2013.					
Date of Session: October 9 and October 10, 2012 (October 2012 Partnering Meeting)					
Scoping Session Purpose: Sampling and Analysis Plan Development and Site Walkover					
Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
Harvey Pokorny	RPM	NAVFAC MW	(847) 688-2600 x611	harvey.pokorny@navy.mil	NAVFAC MW RPM
Val Jurka	Environmental Engineer	NAVFAC LANT	(757) 322-8319	val.jurka@navy.mil	Technical Advisor
Stephanie Warino	Geoscientist	Tetra Tech NUS Inc.	(412) 921-8868	stephanie.warino@tetrattech.com	Outgoing Navy Consultant
Paul Walz	Project Engineer, PM	Bay West	(651) 291-3491	paulw@baywest.com	Navy Consultant for Operation and Maintenance and Sampling
Sheila Desai	RPM	USEPA	(312) 353-4150	desai.sheila@epa.gov	USEPA RPM
Karla Brasaemle	Senior Geologist	Tech Law	(415) 762-0566	kbrasaemle@techlawinc.com	USEPA Consultant
Nicole Goers	USEPA Consultant	Tech Law	(540) 836-0420	ngoers@techlawinc.com	USEPA Consultant/Meeting Minutes
Deepa de Alwis	PM	MPCA	(651) 757-2572	deepa.dealwis@state.mn.us	MPCA PM
Dean Krebs	MPCA Consultant	Antea	(651) 697-5243	dean.krebs@anteagroup.com	MPCA Consultant
Paul Lucas	MPCA Consultant	Antea	(651) 697-5192	paul.lucas@anteagroup.com	MPCA Consultant
Debra Kraemer	Facilitator	The Management Edge	(813) 254-4535	kraemerd@tampabay.rr.com	Partnering Meeting Facilitator
Ken Brown	PM	Resolution Consultants	(414) 944-6192	kenneth.brown@aecom.com	CLEAN Program Oversight
Chris Boehm Carlson	PM	Resolution Consultants	(612) 803-4845	chris.boehm@aecom.com	Navy Consultant CTO PM



Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
Scott Tarmann	Project Engineer	Resolution Consultants	(414) 944-6192	scott.tarmann@aecom.com	Navy Consultant Project Engineer
Cathy Larson	PC	Resolution Consultants	(763) 551-2474	cathy.larson@aecom.com	Navy Consultant PC

Notes:

- CLEAN = Comprehensive Long Term Environmental Action Navy
- CTO = Contract Task Order
- MPCA = Minnesota Pollution Control Agency
- NAVFAC = Naval Facilities Engineering Command
- NAVFAC LANT = Naval Facilities Engineering Command, Atlantic
- NAVFAC MW = Naval Facilities Engineering Command, Midwest
- PC = Project Chemist
- PM = Project Manager
- RPM = Remedial Project Manager
- USEPA = United States Environmental Protection Agency

October 2012 Partnering Meeting Comments/Decisions: Prior to the scoping session, NAVFAC MW provided the Partnering Team with an internal preliminary draft of the SAP for discussion. Comments were provided by USEPA and MPCA, and the SAP and comments were discussed by the Partnering Team.

October 2012 Partnering Meeting Action Items: To aid in identifying specific boring locations, Resolution Consultants requested an as-built of the building in the area of the proposed borings from NAVFAC MW. Resolution Consultants will move forward with preparation of the draft SAP for the Source Area Investigation.

October 2012 Partnering Meeting Consensus Decisions:

The following approach was generally agreed to for the development of the SAP:

1. Two mobilizations will be used for the membrane interface probe (MIP) borings. During the first mobilization, 20 borings will be advanced. MIP results obtained will be provided to the NAVFAC MW Remedial Project Manager (RPM) who will provide results to USEPA and MPCA for input on the locations of the remaining eight MIP borings during the second mobilization.
2. During a third mobilization, up to 28 soil borings will be advanced adjacent to the original MIP borings. Hydraulic conductivity readings will be collected continuously during boring advancement. Groundwater samples will be collected from these borings at approximately four depths. One depth will be at 7 feet below the groundwater table. Other depths will be selected based on the MIP results.
3. A fourth mobilization will be performed to install three groundwater monitoring wells. Groundwater sample results and MIP results will be reviewed to determine the optimum location for the three planned wells. Rather than complete a well nest with shallow,



intermediate, and deep screened intervals, three individual wells at the intermediate depth will be installed. Well screen depth, however, is dependent upon investigation results.

4. During the advancement of the MIP borings, clay layers will be penetrated. To minimize the potential for contaminated groundwater to migrate vertically, soil borings will be abandoned immediately after advancement and groundwater sample collection.
5. If refusal occurs during drilling, a second attempt will be made adjacent to the same location. If refusal occurs a second time, a field decision shall be made regarding further attempts to drill at that location.
6. Both equipment used for the MIP screening and the drilling equipment should have the capability of advancing to 100 feet below ground surface (bgs).
7. The following procedures for verifying the MIP results were discussed:
 - The MIP shall be calibrated daily at the start, middle, and end of day, in accordance with the manufacturer's instructions. If middle or end of day calibration fails, the Project Team will determine if any borings will be redone.
 - MIP results will be compared to groundwater sample results to evaluate the correlation between the two data sets.
 - No soil samples will be collected for comparison to the MIP results. The technology used to advance both the MIP and the borings for groundwater sampling do not allow the option for collecting soil samples. This approach was modified following the January 2013 meeting as described below.
8. The possibility of collecting soil vapor samples during the drilling associated with this study was discussed. The conclusion was to look into this further during future discussions for two reasons: 1) the cost savings of doing both studies together was not considered substantial; and 2) further planning would be required to adequately address the data needs and objectives for soil vapor samples in relation to building parameters.
9. The need for sampling microorganisms in either soil or groundwater shall be evaluated during the development of the SAP.
10. Procedures for performing a field-scale trial of emulsified zero valent iron (EZVI) will be addressed in a separate work plan. (Note: To address Item #9 above, it was decided to look at microbe assays during development of the EZVI injection work plan, to be submitted under separate cover.)
11. Although groundwater sampling and analysis procedures are included in this SAP, this SAP is not intended for use for the annual monitoring, since both sampling and analytical contractors are different.



January 2013 Partnering Meeting Action Items: Following a meeting between the NIROP site developer, Navy, MPCA, USEPA, and the City of Fridley, it was agreed that the SAP should be modified to include assessment of vadose zone soil conditions and a more comprehensive assessment of groundwater quality. Based on this, the source investigation scope of work has been modified as noted below:

1. Perform quantitative assessment of vadose zone soil conditions not previously investigated through collection of continuous soil samples in the vadose zone at soil boring locations near the East Plating Room with four laboratory analytical samples from each boring for volatile organic compound (VOC) analysis.
2. Improve the physical characterization of subsurface soil conditions by conducting soil sampling in the vertical profile borings.
3. Improve understanding of groundwater trichloroethene (TCE) distribution by expanding the groundwater screening program to include 27 vertical profile borings with collection of groundwater samples at 5-foot intervals in granular zones below the water table for VOC analyses.
4. Limit the application of MIP to eight borings near the East Plating Room based on MIP VOC sensitivity of approximately 1 part per million (ppm).



SAP WORKSHEET #10: PROBLEM DEFINITION

(UFP-QAPP Manual Section 2.5.2)

This worksheet presents general background information and the current conceptual site model (CSM), which serves as the basis for developing the sampling and analysis program for the NIROP source area investigation.

10.1 Introduction

The primary problem is that TCE and associated degradation products are present in soil and groundwater beneath the NIROP building. A groundwater extraction and treatment system is in place and contains the impacted groundwater, but it will take a long time for the groundwater to reach cleanup goals at the current pace as source areas continue to dissolve TCE and its degradation products into groundwater at elevated concentrations. The groundwater extraction and treatment system does little to address this condition.

Within this context, the Navy has elected to collect data needed to evaluate if source control remediation could accelerate cleanup of the facility. If warranted, this may result in bench scale testing to evaluate in-situ remedial options that may accelerate cleanup of the facility. More specifically, additional data is needed to:

1. Determine the concentrations and vertical distribution of impacts in the presumed East Plating Room source area.
2. Determine the current shallow groundwater concentrations and vertical distribution of TCE impacts in the presumed Area of Concern (AOC)-17 source area and assess if there are impacts between AOC-17 and MS-31.
3. Determine if there is a connection between the elevated concentrations detected at monitoring well MS-33I and the East Plating Room presumed source area.
4. Evaluate groundwater flow paths from the Paint Shop and determine if they extend onto the NIROP site and are captured by the NIROP extraction wells.
5. Assess groundwater concentrations of TCE and associated degradation products downgradient from source areas.
6. Evaluate the vadose zone soil concentrations of TCE and associated degradation products in the East Plating Room area.



7. Estimate the baseline groundwater concentrations of total and dissolved iron, sulfate, nitrate, methane, ethane, and ethene in groundwater near the source areas to aid in evaluating remedial alternatives.
8. Determine the general soil types and baseline concentrations of total organic carbon (TOC), iron, and TCE along with its associated degradation products in the source areas.

The purpose of the project is to define the nature and contaminant concentrations within the subsurface soil and groundwater at the potential source areas and downgradient areas and to collect data needed for designing potential future remedial actions. Based upon results of previous investigations, the presumed primary source area appears to be beneath the East Plating Room, which will be a primary focus area for this investigation. Secondary source areas to be investigated are AOC-17 in the northwest portion of the building (a former wash rack sump), and the area east and north of monitoring well MS-33I (referred to as 7th and Broadway). The area downgradient of the BAE Systems (BAE) Paint Shop will also be investigated. Figure 10-1 illustrates the site layout and features. For reference, a site location map and a site plan, from previous reports, are included in Appendix A (Figures A-1 and A-2). A CSM figure outlining exposure pathways, taken from the Record of Decision (ROD) for OU2 and OU3, is also included in Appendix A (Figure A-3).

The NIROP facility is divided into three separate OUs for ease of addressing its contaminant issues:

- OU1 is the groundwater beneath the entire property,
- OU2 is the unsaturated soils outside of the building footprint on the NIROP property,
- OU3 is the unsaturated soils beneath the NIROP building and saturated soil either under or outside the building.

The investigation outlined in this SAP involves groundwater and saturated soil beneath the building and outside the building which is part of OU1 and OU3. This investigation also includes unsaturated soil beneath the building which is part of OU3. No investigation activities are planned for OU2. Up to five contingency borings are included as part of this project and will be used as needed, in consultation with the USEPA and MPCA, to address data gaps.

The results of this investigation will be used to evaluate in-situ remedial options and the need for bench scale testing. Bench scale test results would, in part, be used to evaluate the degree to which remediation could be accelerated, although it should be noted that the degree of acceleration will be approximate in nature. Bench scale testing is not part of the effort for this project.

A second problem is that concentrations of TCE and associated degradation products in shallow soil above the water table have not been adequately characterized near the East Plating Room. Additional data is needed to determine appropriate actions during proposed site redevelopment activities.

10.2 The Environmental Questions Being Asked:

- What are the concentrations and vertical profile of source area impacts at the East Plating Room?
- What are the concentrations and vertical profile of source area impacts at AOC-17?
- Is there a connection between the elevated concentrations detected at MS-33I and the East Plating Room?
- Is the Paint Shop source area contributing to groundwater impacts on the NIROP site and in the NIROP extraction wells?
- What are the concentrations and vertical profile of TCE and associated degradation products in groundwater downgradient from the source areas?
- What are vadose zone concentrations of TCE and associated degradation products in shallow soil at the East Plating Room area?
- What are the baseline concentrations of total and dissolved iron, sulfate, nitrate, methane, ethane, and ethene in groundwater near the East Plating Room areas?
- What are the general soil types and baseline concentrations of TOC, iron, and TCE along with its associated degradation products in the source areas?

10.3 Observations From any Site Reconnaissance Reports:

Site maps, surrounding site information, and specific site details are found in the following documents:

- Record of Decision for Groundwater Remediation, Naval Industrial Reserve Ordnance Plant Fridley, Minnesota, September 28, 1990; filed in the NIROP Facility Administrative Record as N91192_000078. USEPA.
- Remedial Investigation for Operable Unit 3, Naval Industrial Reserve Ordnance Plant Fridley, Minnesota, April 2002. Tetra Tech.
- Record of Decision for Operable Unit 2 and Operable Unit 3, Naval Industrial Reserve Ordnance Plant Fridley, Minnesota, August 2003; filed in the NIROP Facility Administrative Record as N91192_000661. USEPA.



- 2011 Annual Monitoring Report, Naval Industrial Reserve Ordnance Plant Fridley, Minnesota, November 2012. Tetra Tech.
- Source Investigation Map Methodology NIROP Superfund Site Fridley, Minnesota, February 2012. NAVFAC.
- Technical Memorandum "Discrete Groundwater Sampling Results, Naval Industrial Reserve Ordnance Plant" Fridley, Minnesota, August 31, 2012. CH2M HILL.
- Figure 10-1 of this SAP.

Previous site investigations and annual monitoring reports have documented the general extent of the groundwater plume. A groundwater extraction and treatment system is in operation to minimize the flow of contaminated groundwater from the NIROP. The focus of this investigation is additional evaluation of suspect source areas beneath the building. Details regarding the most recent evaluation of the source areas are presented below.

10.3.1 A Synopsis of Secondary Data or Information from Site Reports

NAVFAC MW conducted a data review of the potential source areas beneath the building and results were presented in "Source Investigation Map Methodology NIROP Superfund Site" dated February 2012. The objective of the data review was to ascertain if data obtained in 1995 and 1997 was sufficient to delineate source area(s) beneath the NIROP complex building. A figure depicting the potential source areas was constructed using TCE groundwater concentration data from all groundwater depths, incorporating maximum observed concentrations. This figure is included for reference in Appendix A (Figure A-4). Observation of the plotted data suggests four potential source areas:

- East Plating Room;
- AOC-17 (a former wash rack sump);
- Area east and north of monitoring well MS-33I; and
- BAE Paint Shop Resource Conservation and Recovery Act (RCRA) Site (located south of Navy controlled property).

Soil data from 1995 and 1997 were also reviewed and results point to the East Plating Room and 7th and Broadway as major sources of the TCE release. It is inferred that the dense solvent migrated downward and saturated the fine-grained stratigraphic unit(s). The highest TCE soil concentrations were recorded within or adjacent to fine-grained materials. Smaller source areas



were also revealed in the northwest quadrant of OU3 (near AOC-17). Based upon more recent data collected after the Remedial Investigation (RI), it appears that dispersion from a BAE RCRA Paint Shop source area (south of Navy-controlled property) may have affected groundwater beneath OU3. As discussed in the October 12, 2012 meeting, VOCs were very low or were not detected in sandy samples.

10.3.2 Site Walkover Findings

Potential boring locations at the East Plating Room, the AOC-17 area, and the area east and north of MS-33I were observed for access issues, potential ventilation concerns, and concrete thickness. The floor of the East Plating Room included an area that was a recessed pit approximately 8 feet deeper than the surrounding slab on grade. The pit below the East Plating Room was filled in with sand and concrete and is now level with surrounding grade. Due to access limitations from BAE operations in the East Plating Room and concerns over penetrating the concrete floor at the bottom of the pit (which is part of the OU3 remedy outlined in the ROD), borings completed as part of this investigation will be completed outside and adjacent to the East Plating Room.

An as-built type map was requested to aid in placing the source area investigation borings and was used as the base map for Figure 10-1.

The building currently is used by BAE, engaged in the development, delivery, and support of advanced defense, security and aerospace systems. Entry into the building requires security clearance. Security requirements will be confirmed before initiating on-site work, but it is assumed that confirmation of U.S. citizenship is required for all on-site personnel to work at the site.

10.4 The possible classes of contaminants and the affected matrices:

TCE and its degradation products are the VOCs of concern for this investigation. The selected VOC list for the project is the list identified in the 2011 Annual Monitoring Report and includes: 1,1-dichloroethane, 1,1-dichloroethene, cis-1,2-dichloroethene, trans-1,2-dichloroethene, tetrachloroethene, 1,1,1-trichloroethane, TCE, and vinyl chloride.

Affected matrices are unsaturated soil (vadose zone), saturated soil (beneath water table), and groundwater.

10.5 The rationale for inclusion of chemical and nonchemical analyses:

This investigation will produce both chemical and nonchemical analysis. The MIP investigation will produce nonchemical data used to model the relative concentrations of VOCs in the subsurface and the soil types based on the electrical conductivity (EC) log (fine grained vs. coarse-grained materials). The MIP tool was selected because it is an efficient and cost effective way to screen for



VOCs, and the modeling performed on the MIP data can be very illustrative of site conditions. The MIP data will also supplement the CSM, and will aid in designing future remedial actions.

The vertical profile borings will also produce nonchemical data needed to determine the lithology of the subsurface materials. This data is needed to assist with identifying preferential flow paths and the nature of the source areas. During advancement of the vertical profile borings, groundwater samples will be collected for chemical analysis for correlation with the MIP data, to determine the distribution of contaminants, and to supplement the CSM. The groundwater samples collected from the monitoring wells will include chemical analysis to help assess remedial options, monitoring potential pilot test results, and supplement the monitoring well network at the site. Direct chemical analysis is the only acceptable methodology for comparison to regulatory framework.

10.5.1 Membrane Interface Probe

The MIP provides qualitative VOC screening data using electron capture detector (ECD), halogen specific detector (XSD), flame ionization detector (FID), and photoionization detector (PID). According to Vironex, Inc., the ECD instrument provides detection levels as low as 0.5 ppm in the field. According to Vironex, Inc., the MIP has been used in areas with TCE nonaqueous phase liquid (NAPL) without reaching upper detection limit of the XSD instrument. The MIP will be used as a reconnaissance tool to assess the East Plating Room area. The MIP also has an EC dipole on the probe which provides an indication of soil particle size. The MIP works in both the saturated and unsaturated zones and in clays, sands, and gravels.

Borings will be advanced to approximately 80 feet bgs and continuously screened with the MIP. The MIP results will be used to create a three dimensional visualization of soil types and contaminant concentrations near the East Plating Room to help illustrate potential source areas and downgradient flow pathways. Two dimensional cross-sections will be presented in the report.

10.5.2 Analysis of Soil and Groundwater in Vertical Profile Borings

Soil and groundwater conditions will be evaluated at 27 locations beneath the facility. The objectives of the vertical profile borings are to:

- Understand the hydrostratigraphy with particular focus on the locations of low and high permeability units within the flow system.
- Understand the distribution and concentrations of VOCs dissolved in groundwater within transmissive zones.
- Provide baseline concentrations of VOCs for evaluating potential source area remedial options.



- Provide analytical results that can be used to correlate the accuracy of MIP results.
- Provide vadose zone soil VOC concentrations near the East Plating Room to support site redevelopment activities.

At each vertical profile location, two side-by-side borings will be advanced. In the first boring, soil samples will be continuously collected to the bottom of the boring (80 feet). These samples will enable characterization of subsurface formations and allow for identification of likely preferred flow paths and fine grained, low permeability conditions. Vadose zone soil samples will be collected from select borings near the East Plating Room for VOC analysis. Soil samples may be collected from beneath the water table for VOC analysis in locations where obtaining a groundwater sample would be impracticable (e.g., silt/clay units greater than 5 feet thick). Soil samples from approximately 10 locations will also be analyzed for TOC and iron to establish baseline site concentrations for use in remedial selection/design. In the second side-by-side boring, groundwater samples will be collected with a temporary well screen at approximately 5-foot intervals for laboratory analysis of VOCs. The 5-foot sample intervals will allow for a more detailed review of the distribution of VOCs and a refined interpretation of potential flow pathways. All of the proposed soil borings will be completed and sealed, followed by completion of borings for groundwater sample collection.

10.5.3 Analysis of Soil and Groundwater at Monitoring Wells

Three monitoring wells will be installed at locations to be selected following review of the MIP and vertical profiling data.¹ Soil samples will be collected at monitoring well locations for VOC analysis to provide baseline concentrations for evaluating remedial options. At least 2 weeks following well development, groundwater samples will be collected from the monitoring wells using low flow sampling methods and analyzed for VOCs. The VOC data will be used to:

- Correlate the MIP and vertical profile results;
- Provide baseline data to evaluate remedial options;
- Supplement the existing monitoring well network; and
- Provide monitoring points to evaluate the effectiveness of any remediation pilot tests.

¹ If well installation is required within the building, appropriate ventilation measures will be taken, as described in the Health and Safety Plan, and proper well construction measures will be implemented to control the potential for vapor intrusion, as described in Section 17.5.2 of this SAP.



Soil samples will also be analyzed for TOC and iron. Groundwater samples from the new monitoring wells will also be analyzed for sulfate, nitrate, total and dissolved iron, and methane, ethane, and ethene to establish baseline site concentrations for use in remedial selection/design.

10.5.4 Investigative-Derived Waste

Soil and liquid samples representative of the investigative-derived waste (IDW) will be analyzed in accordance with the disposal acceptance criteria as specified from the disposal facility. The analyses will likely include toxicity characteristic leaching procedure (TCLP)-VOCs, TCLP-semivolatile organic compounds (SVOCs), TCLP-metals, TCLP-pesticides, polychlorinated biphenyls (PCBs), paint filter test, pH, and ignitability, as required by the disposal firm, to properly profile waste for disposal. Analyses will be performed in accordance with requirements in 40 Code of Federal Regulations (CFR) 261, Subpart C, and further information regarding data quality will not be specified.

10.6 Information concerning various environmental indicators

The present condition of the environment is outlined in the 2011 Annual Monitoring Report, and the previously referenced soil and groundwater investigation reports. The CSM was presented in the ROD for OU2 and OU3, and is included in Appendix A (Figure A-3). The data gathered during this effort may be used to update and revise the CSM. The data collected as part of this investigation will also be used as a baseline for evaluating the effectiveness of any future remedial actions.



SAP WORKSHEET #11: PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

(UFP-QAPP Manual Section 2.6.1)

11.1 Who Will Use the Data?

The Project Team, which consists of the Navy, USEPA Region 5, MPCA, and Resolution Consultants, will use the data to assess the general magnitude of source area contamination and vertical pathway distribution prior to capture. When complete, data will also be available for presentation to the general public to provide status information regarding progress of the site cleanup.

11.2 What Will the Data be Used For?

The data will be used by the Project Team to characterize the concentration and vertical distribution of VOCs in the potential source areas and to better understand the downgradient distribution in groundwater. The data will be used to evaluate remedial methods that may be useful in reducing the concentrations in source areas to levels that will allow regulatory control/closure in an accelerated timeframe that is acceptable to the project team. The degree of cleanup time acceleration will be evaluated following bench scale testing.

11.3 Problem Statement

The primary problem is that TCE and associated degradation products are present in soil and groundwater beneath the NIROP building. A groundwater extraction and treatment system is in place and contains the impacted groundwater, but it will take a long time for the groundwater to reach cleanup goals at the current pace as source areas continue to dissolve TCE and its degradation products into groundwater at elevated concentrations. The groundwater extraction and treatment system does little to address this condition. There is currently insufficient data to adequately fully characterize the source areas or to fully understand the downgradient distribution of TCE and its degradation products. Additional data is needed to evaluate remedial methods that may be useful in reducing the concentrations in source areas to levels that will allow regulatory control/closure in a timeframe that is acceptable to the Navy.

A second problem is that concentrations of TCE and associated degradation products in shallow soil above the water table have not been adequately characterized near the East Plating Room. Additional data is needed to determine appropriate actions during proposed site redevelopment activities.

11.4 Goals of the Study (If...,Then Statements)

The goals of the study are to:



1. Determine the concentrations and vertical distribution of impacts in the presumed East Plating Room source area.
2. Determine the current shallow groundwater concentrations and vertical distribution of TCE impacts in the presumed AOC-17 source area and assess if there are impacts between AOC-17 and MS-31.
3. Determine if there is a connection between the elevated concentrations detected at monitoring well MS-33I and the East Plating Room presumed source area.
4. Evaluate groundwater flow paths from the Paint Shop and determine if they extend onto the NIROP site, and are captured by the NIROP extraction wells.
5. Assess groundwater concentrations of TCE and associated degradation products downgradient from source areas.
6. Evaluate the vadose zone soil concentrations of TCE and associated degradation products in the East Plating Room area.
7. Estimate the baseline groundwater concentrations of total and dissolved iron, sulfate, nitrate, methane, ethane, and ethene in groundwater near the source areas to aid in evaluating remedial alternatives.
8. Determine the general soil types and baseline concentrations of TOC, iron, and TCE along with its associated degradation products in the source areas.

If the investigation results indicate the definitive presence of a source area with conditions favorable for remediation, then remedial options will be evaluated and a work plan for bench scale testing, likely consisting of injection of EZVI, will be prepared.

If no clear treatable source is identified, then results will be reviewed to determine if further investigation could be used to identify treatable source material, or if contamination has dispersed to a point where alternate remediation techniques may be preferred.

If subsurface conditions limit the depths achievable using the recommended direct sensing equipment and direct push drilling techniques, then the drilling approach may be modified to include larger drill rigs, modified direct sensing equipment, or a change in the overall scope of work.

11.5 Inputs to Problem Resolution

The inputs needed to achieve the projects goals identified in Section 11.4 include field observations and measurements and chemical data as described below.



- The following field screening data will be used to help select the locations and depths of laboratory analytical samples and aid in defining the potential source areas and subsurface lithology:
 - VOC screening data will be gathered from MIP borings from 0 to approximately 80 feet bgs to aid in defining VOC source areas in the East Plating Room area.
 - While advancing MIP borings, EC data will be collected to aid in indentifying general soil types. This information will help identify layers that would be more likely to have elevated concentrations of TCE (low permeability layers) and groundwater flow pathways (high permeability layers).
 - Logging of vertical profile boring soil cores by a trained geologist and field screening soil with a PID will provide information on zones with elevated VOC concentrations and zones that may represent potential source areas.
- Purge water generated prior to groundwater sample collection from monitoring wells will be field screened using a YSI water quality meter (or equivalent) and Hanna turbidity meter (or equivalent) to determine when formation water is being produced and a representative groundwater sample can be collected. Field screening parameters will include temperature, pH, dissolved oxygen (DO), specific conductance, oxidation-reduction potential (ORP), and turbidity. SOP 3-24 will be followed (Appendix B).
- Soil and groundwater samples will be collected for off-site laboratory analysis following SOPs in Appendix B from the following locations:
 - Groundwater samples will be collected from up to 27 vertical profile borings at up to 12 depths in each boring (approximately every 5 feet) using a temporary screen point sampler. Groundwater sampling results will be used to investigate the distribution of VOCs both vertically and horizontally and to correlate the MIP results and PID results. The groundwater results will be considered when determining the location and screen depth of the long-term monitoring wells. Groundwater samples will be collected generally following the procedures in SOP 3-14 using a bladder pump and analyzed for VOCs.
 - In the vertical profile borings, soil samples will be collected beneath the water table in lieu of groundwater samples where low permeability (e.g., silt or clay) units greater than 5 feet thick are encountered. The most impacted portion of the soil core will be selected for laboratory analyses. Selection will be based on a combination of careful PID screening, MIP, and geologic interpretation. The soil sample will be analyzed for VOCs, and the results will be used to characterize potential source areas beneath the water table where groundwater samples cannot be effectively collected using temporary well screens. Approximately five soil samples will also be analyzed for TOC and iron to provide data for evaluating remedial options. Soil samples will be collected from the direct push drill rig soil core following the procedures in SOP 3-21 (Appendix B).



- Vadose zone soil samples will be collected from nine soil borings (combination of vertical profile borings and shallow vadose zone borings near East Plating Room) with four analytical samples per boring for VOC analysis (a sample approximately every 5 feet). Approximately five soil samples will also be analyzed for TOC and iron. The soil sampling results will be used to characterize vadose zone soil conditions. Soil samples will be collected from the direct push drill rig soil core following the procedures in SOP 3-21 (Appendix B).
- Nine additional soil samples will be collected from the three monitoring well soil borings (three samples per monitoring well boring). Soil samples will be analyzed for VOCs to assess the extent of VOC impacts. Soil samples will also be analyzed for TOC and iron. Soils samples will be collected from the sonic drill rig soil core following the procedures in SOP 3-21 (Appendix B).
- Groundwater samples will be collected from the three newly installed monitoring wells and analyzed for VOCs. Results will be used to provide baseline concentrations if source area treatment is performed. Additionally, samples from the three newly installed wells will be analyzed for sulfate, nitrate, total and dissolved iron, methane, ethane, and ethane to provide data for evaluating remedial options. Groundwater samples will be collected following procedures in SOP 3-14 (Appendix B).
- Up to five contingency borings will be added to the subsurface investigation program to address data gaps within the results of the field program. The need for contingency borings will be discussed with the USEPA and MPCA during conference calls at the conclusion of the MIP and vertical profile boring programs. Criteria used to select locations for contingency borings include conflicting data results between boring locations, anomalous data results, and/or other unexpected conditions. Contingency boring locations, sampling intervals, media, and laboratory analytical schedule will be discussed with the USEPA and MPCA prior to implementation.
- One soil and one water IDW sample will be collected and analyzed for TCLP-VOCs, TCLP-SVOCs, TCLP-metals, TCLP-pesticides, PCBs, ignitability, pH, and paint filter test, as required by the disposal firm. IDW samples will be collected following procedures in SOP 3-21.
- Quality control (QC) data, including field duplicates, equipment blanks, and matrix spikes (MS)/matrix spike duplicates (MSDs) will be collected to aid in evaluating data quality. Data validation will be performed using the QC results.
- Land survey horizontal and vertical control will be obtained for all soil borings and monitoring wells. The survey will be completed by a registered land surveyor (Harry S. Johnson Co).



11.6 Define the Study Boundaries

The site investigation areas include the primary source area which is considered to be the East Plating Room area. Secondary source areas to be investigated are AOC-17 in the northwest portion of the building (a former wash rack sump), and the area east and north of monitoring well MS-33I. The area between the groundwater extraction wells and potential source areas will also be assessed to help define groundwater flow pathways. Figure 10-1 illustrates the site layout and features and the locations of investigation areas.

The vertical boundary extends from the ground surface to approximately 80 feet bgs; however, borings may need to be advanced deeper in some locations in order to encounter the targeted confining layer. Drilling to these depths will reach the practical limits of the drilling equipment and refusal may occur prior to reaching the lower confining layer.

The temporal boundaries for this study will be the period of the actual field investigation, anticipated to occur from July through December 2013. The majority of the work will be completed indoors. There are no seasonal variations anticipated to affect this investigation.

11.7 Analytical Approach

Specific analytes and their respective project action limits (PALs) are provided in Worksheet #15. Regulatory sources for these PALs are as follows:

Media	Reference for PALs
Groundwater	The United States Environmental Protection Agency Maximum Contaminant Levels found at: http://water.epa.gov/drink/contaminants/index.cfm
	The lowest value on the Minnesota Department of Health Human Health-Based Water Guidance Table found at: http://www.health.state.mn.us/divs/eh/risk/guidance/gw/table.html
Soil	The Minnesota Pollution Control Agency (MPCA) Industrial Soil Reference Value are found at: http://www.pca.state.mn.us/index.php/waste/waste-and-cleanup/cleanup-programs-and-topics/topics/risk-based-site-evaluation-process-guidance-documents.html?menuid=&missing=0&redirect=1
	The MPCA Soil Leachate Values are found at: http://www.pca.state.mn.us/index.php/view-document.html?gid=3152
Waste	Code of Federal Regulations 261.24

For groundwater, the lowest of either the maximum contaminant level (MCL) or the Minnesota Department of Health (MDH) criteria will be the PAL. For soils, the industrial Soil Reference Values (SRVs) were selected for comparison because the site is currently zoned industrial. The current operations are industrial, and it is understood that future land use will remain commercial/industrial as part of the institutional control outlined in the 2003 ROD.

11.8 Performance or Acceptance Criteria

The objective of this section is to complete the following:

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

Sampling Strategy — Both a judgmental and phased approach were used in developing the sampling design. Initial sampling locations are biased to areas where historical data indicates elevated TCE concentrations. Subsequent sample locations will be selected using a phased approach to optimize sample locations by using information obtained during prior phases.

During the course of the project, interim results will be provided by Resolution Consultants to the Navy RPM for distribution to the Project Team to obtain feedback. Interim results will be presented as follows:

- After completion of the eight MIP borings, the MIP logs will be presented to the Project Team.
- After completion of the 27 vertical profile borings and three vadose zone borings, the result will be presented to determine if the five contingency borings will be needed to address data gaps. Contingency borings may be used to collect shallow vadose zone soil samples, deep soil samples for geologic characterization, or groundwater vertical profile samples.
- Prior to installation of the three monitoring wells, results of the field program and recommendations for the location and screen position of the three monitoring wells be presented to the project team to gain consensus.

Sources of Error — Sources of error may be divided into two main categories: sampling errors and measurement errors. A sampling error occurs when the sampling design, planning, and/or implementation do not provide data representative of site conditions. A measurement error occurs because of performance variance from laboratory instrumentation, analytical methods, and operator error. The USEPA identifies the combination of all these errors as a “total study error” (USEPA 2006). One objective of the investigation is to reduce the total study error so that decision-makers can be confident that the data collected accurately represent the chemical characteristics of the site.



Managing Decision Error — The investigation will utilize decision-error minimization techniques in sampling design, sampling methodologies, and laboratory measurements. 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 and #21).
- Use applicable analytical methods and SOPs for sample analysis by a competent analytical laboratory with a current MDH certification and a Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) certification.
- Confirm analytical data to identify and control potential laboratory error and sampling error by using spikes, blanks, and replicated samples.

Field Measurements — The following acceptance criteria will be used for field measurements:

- Field screening TCE concentrations may range from greater than 10 ppm in source areas to less than 0.5 ppm in areas less impacted by TCE. The MIP VOC results need to be of sufficient quality to distinguish the different conditions along this range of concentrations. This wide range of detections will be accomplished through the use of two instruments: the XSD and ECD.
- Field screening EC data to determine relative grain size needs to be of sufficient quality to distinguish between high electrically conductive soil types (silts and clays) and low conductivity soil types (sands and gravels) so that the groundwater samples can be collected at the proper locations and to support the CSM.
- Field screening of the groundwater parameters is a standard procedure in the development and sampling of wells. Field screening of the groundwater parameters shall be of sufficient quality to determine whether the aquifer has stabilized so that samples collected represent actual aquifer characteristics.
- Land survey data will be conducted by a licensed land surveyor licensed in the State of Minnesota. The survey horizontal control will be referenced to the World Geodetic System of 1984 (World Geodetic System of 1984) datum, with horizontal coordinates provided as latitude and longitude pairs. Horizontal positions will be measured to within 0.5 meter accuracy. Vertical control will be North American Vertical Datum 1988 with elevations reported in feet. The top of concrete elevation at each boring will be measured to an accuracy of 0.1 feet; however the elevation of monitoring well riser casings will be measured to an accuracy of 0.01 feet. The survey may be conducted using both global positioning system and Total Station techniques.



Field Data Logs — All sample information will be transcribed into a field logbook and/or onto field data sheets.

Analytical Laboratory Sample Management — The sample matrix, number of samples, and number and type of laboratory QA/QC samples are summarized in Worksheets #18, #19, #20, and #30. Also included are details on the analytical group, sample volumes, sample container specifications, preservation requirements, and holding times.

The laboratory will provide electronic data deliverable files, portable document format files of the data deliverables for all project data, and a hard copy of data deliverables for all results. Designated samples will be used to obtain necessary subsamples for laboratory QC measurements (i.e., analytical sample duplicate and sample MS/MSD). Tasks will be completed using the laboratory SOPs.

Resolution Consultants will provide data validation services and verify and evaluate the usability of the data as described in Worksheets #34 through #36.

Portable document format copies of all analytical data packages will be stored on CD-ROM, archived in the Naval Facilities, Atlantic Administrative Record, and uploaded onto the Naval Installation Restoration Information Solution (NIRIS) system. All other data generated in the field and reports generated for the project will be stored as computer readable data files by Resolution Consultants.

The primary goal of the project is to define the source areas for TCE under the NIROP building. Thus, samples are being collected in areas where high concentrations of TCE and its degradation products are expected to be encountered. These high concentrations have the potential to contaminate the analytical instrumentation if analyzed undiluted. To avoid this, the laboratory will provide results for VOCs within the instrument calibration range, and, if not-detected, at a limit of quantitation (LOQ) less than the PAL for water samples and the industrial SRV for soil samples. However, in cases where high concentrations of TCE or other VOCs are present, the PALs may not be achievable. In these cases, the laboratory will use the lowest dilution possible to report TCE and other high concentration VOCs within the instrument calibration range. Following a successful quantitation of these high analytes, the laboratory will then run a second lower dilution at 10 times less than the highest dilution required to quantify all other VOCs. Soil LOQs listed will also be adjusted for moisture content.

The PAL for vinyl chloride in water (0.2 microgram per liter ($\mu\text{g/L}$)) is less than the proposed laboratory limit of detection (LOD) of 0.5 $\mu\text{g/L}$. This LOD is considered acceptable because the goal for the project is to identify TCE source material to allow evaluation of potential remedial actions.



The focus of the project is to identify areas of high TCE concentration (above approximately 500 µg/L) rather than to delineate areas to the PALs, which was already completed as part of the RI. Vinyl chloride results will be used to provide information regarding current concentrations for this TCE breakdown product and its location throughout the investigation area. Non-detects will be highlighted in the final report to indicate that the LOD is not less than the PAL so that areas of non-detect are not mistakenly interpreted as clean. Vinyl chloride is included in the annual monitoring program with an LOD less than the PAL; thus, groundwater is being monitored through alternative studies at concentrations to be protective of human health.

If the investigation results indicate the definitive presence of a source area with conditions favorable for remediation, then remedial options will be evaluated and a work plan for bench scale testing, likely consisting of injection of EZVI, will be prepared.

If no clear treatable source is identified, then results will be reviewed to determine if further investigation could be used to identify treatable source material or if contamination has dispersed to a point where alternate remediation techniques may be preferred.

If subsurface conditions limit the depths achievable using the recommended direct sensing equipment and direct push drilling techniques, then the drilling approach may be modified to include larger drill rigs, modified direct sensing equipment, or a change in the overall scope of work.

11.9 How "Good" do the Data Need to be in Order to Support the Environmental Decisions?

In order to support the environmental questions being asked, the data needs to be of sufficient quality to meet the performance measures which include precision, accuracy, comparability representativeness, completeness, and sensitivity described in this SAP.

11.10 Data Collection Plan

Data collection will follow a phased approach outlined in the sampling design and rationale presented in Worksheet #17. Data from each phase will be reviewed to identify data gaps and determine if contingency borings are needed. Following the MIP, vertical profile, and contingency borings, the data will be comprehensively reviewed to aide in the selection of monitoring well locations and depths. Sampling procedures are described in site-specific SOPs included in Appendix B. Resolution Consultants will collect or oversee the collection of all field measurements and samples.



MIP screening for VOCs and EC for particle size will be performed by Vironex, Inc. of Golden, Colorado.

Boring advancement and groundwater sample collection using the Geoprobe macro-core soil sampling and temporary screen point groundwater sampling systems will be performed by Matrix Environmental LLC of Maple Grove, Minneapolis.

Groundwater monitoring wells will be installed and developed by Mateco Drilling Company of Rockford, Michigan.

TriMatrix Laboratories in Grand Rapids, Michigan (TriMatrix) will analyze all soil and groundwater samples collected by field personnel.

Boring and monitoring well surveying will be completed by Harry S. Johnson Co of Bloomington, Minnesota.

11.11 Reporting

Results for the project will be presented in the "Source Area Investigation Report for Remedial System Optimization" following the completion of the project tasks included in this SAP. This report will be submitted as draft, draft final, and final.

The report will include:

- A table of contents.
- A schedule of work as completed.
- Methodology.
- Narrative.
- Deviations from the SAP.
- MIP data logs and figures.
- Boring and well logs.
- Three cross sections illustrating MIP data, lithology and vertical profile sample results.



- Site figures including: site plan, cross-sections (with geologic, hydrogeologic, and contaminant distribution), potentiometric surface plan, contaminant distribution plans (soil and groundwater), and other figures if needed to depict site conditions.
- Photographs.
- Analytical data tables comparing results to regulatory criteria (Worksheet #15).
- Testing, transportation and disposal documentation associated with the IDW.
- Data validation results.
- Recommendations.

Revisions to the report will include preparation of a "Response to Comments" memo that itemizes comments received and proposed changes.



SAP WORKSHEET #12: FIELD QUALITY CONTROL SAMPLES

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table — Field QC Samples for Groundwater Samples					
QC Sample	Analytical Group	Frequency	Data Quality Indicators	Measurement Performance Criteria ⁽⁴⁾	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
Trip Blanks	VOCs	One per cooler to the laboratory containing volatiles	Accuracy/Bias/ Contamination/ Representativeness	No analytes >1/2 LOQ, except common lab contaminants, which must be < LOQ	S & A
Field Duplicates from Vertical Profiling Borings	VOCs Field duplicates to be preferentially collected from a consistent interval (at 7 feet below the water table) ⁽³⁾	One per 10 field samples	Precision	Values > 5X LOQ: RPD must be ≤30 ⁽¹⁾	S & A
Field Duplicates from Monitoring Wells	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane/ethene	One per 10 field samples	Precision	Values > 5X LOQ: RPD must be ≤30 ⁽¹⁾	S & A
Equipment Rinsate Blanks	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane/ethene	One per 20 field samples per matrix per sampling equipment ⁽²⁾	Accuracy/Bias/ Contamination/ Representativeness	No analytes >1/2 LOQ, except common lab contaminants, which must be < LOQ	S & A
Matrix Spike/Matrix Spike Duplicate	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane/ethane	One pair per 20 field samples	Accuracy/Bias/ Precision	See Appendix C	S & A
Laboratory Control Samples	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane/ethane	One per preparatory batch	Accuracy	See Appendix C	A
Laboratory Method Blanks	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane/ethane	One per preparatory batch	Accuracy	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. For common laboratory contaminants, no analytes detected >LOQ (see Box D-1 in QSM V4.2).	A
Surrogate Spike Recoveries	VOCs	All field and QC samples	Accuracy	See Appendix C	A



Measurement Performance Criteria Table — Field QC Samples for Groundwater Samples					
QC Sample	Analytical Group	Frequency	Data Quality Indicators	Measurement Performance Criteria ⁽⁴⁾	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
Reporting Limit Verification	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane/ethane	Once per initial calibration	Sensitivity	The lowest standard concentration at or below the laboratory limit of quantitation	A
Cooler Temperature Indicator	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane/ethane	One per cooler	Representativeness	Temperature less than 6 degrees Celsius	S

Notes:

- (1) = If duplicate values are less than five times the LOQ, the absolute difference should be less than or equal to two times the LOQ.
- (2) = Equipment rinsate blanks will be collected if decontamination is required and will not apply if dedicated equipment is used.
- (3) = The interval 7 feet below the water table is being targeted for duplicate sample collection to maintain consistency with the groundwater data collected in 1997. The goal is to obtain a reliable data set at one depth across the site.
- (4) = Control limit provided in Appendix C are those specified in Appendix G of the DoD QSM, Version 4.2, where available.
- DoD QSM = *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2, October 2010, or most recent version at the time of sampling.
- LOQ = limit of quantitation
- QC = quality control
- RPD = Relative percent difference
- VOCs = volatile organic compounds



Measurement Performance Criteria Table — Field QC Samples for Soil Samples					
QC Sample	Analytical Group	Frequency	Data Quality Indicators	Measurement Performance Criteria ⁽²⁾	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
Trip Blanks	VOCs	One per cooler to the laboratory containing volatiles	Accuracy/Bias/Contamination/Representativeness	No analytes > 1/2 LOQ, except common lab contaminants, which must be < LOQ	S & A
Field Duplicates	VOCs, TOC, Iron	One per 10 field samples	Precision	Values > 5X LOQ: RPD must be ≤50 ⁽¹⁾	S & A
Matrix Spike/Matrix Spike Duplicate	VOCs, Iron	One pair per 20 field samples	Accuracy/Bias/Precision	See Appendix C	S & A
Laboratory Duplicate	TOC	One per preparatory batch	Precision	RPD 20%	A
Laboratory Control Samples	VOCs, TOC, Iron	One per preparatory batch	Accuracy	See Appendix C	A
Laboratory Method Blanks	VOCs, TOC, Iron	One per preparatory batch	Accuracy	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. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	A
Surrogate Spike Recoveries	VOCs	All field and QC samples	Accuracy	See Appendix C	A
Reporting Limit Verification	VOCs, TOC, Iron	Once per initial calibration	Sensitivity	The lowest standard concentration at or below the laboratory limit of quantitation	A
Cooler Temperature Indicator	VOCs, TOC, Iron	One per cooler	Representativeness	Temperature less than 6 degrees Celsius	A

Notes:

- (1) = If duplicate values are less than five times the LOQ, the absolute difference should be less than or equal to two times the LOQ.
- (2) = Control limit provided in Appendix C are those specified in Appendix G of the Department of Defense Quality Systems Manual (QSM), Version 4.2, where available.
- LOQ = limit of quantitation
- QC = quality control
- RPD = relative percent difference
- TOC = total organic carbon
- VOCs = volatile organic compounds



**SAP WORKSHEET #13: SOURCES OF SECONDARY DATA CRITERIA AND LIMITATIONS
 TABLE**

(UFP-QAPP Manual Section 2.7)

Secondary Data Criteria and Limitations Table				
Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Grain Size Distribution	<i>A-E Quality Control Summary Report for Well Installation and Groundwater and Soil Sampling at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota (RMT, Inc. 1991) N91192.AR.000079</i>	RMT, Inc.	Basis for estimating the quantity and distribution of emulsified zero valent iron that could be applied in the subsurface during pilot testing and/or full-scale implementation.	None
Prior Source Area Characterization	Source Investigation Map Methodology, February 2012 for summary / interpretation of historic data	Navy	Basis for locating vertical profile borings.	None
Soil and Groundwater Data	Remedial Investigation for Operable Unit 3 Naval Industrial Reserve Ordnance Plant Fridley, Minnesota, April 2002	Tetra Tech Pittsburgh, Pennsylvania	Incorporate into site figures and discussion, as appropriate.	Any limitations cited in the report will be considered limitations for this investigation.
Groundwater Data	2011 Annual Monitoring Report, Naval Industrial Reserve Ordnance Plant Fridley, Minnesota, November 2012	Tetra Tech NUS, Inc. Pittsburgh, Pennsylvania	Incorporate into site figures and discussion, as appropriate.	None
Vertical Distribution of Chlorinated Solvent Locations in Groundwater	Technical Memorandum "Discrete Groundwater Sampling Results, Naval Industrial Reserve Ordnance Plan" Fridley, Minnesota, August 31, 2012	AGVIQ-CH2M HILL Constructors, Inc. Joint Venture III	Incorporate into site figures and discussion, as appropriate.	None



SAP WORKSHEET #14: SUMMARY OF PROJECT TASKS

(UFP-QAPP Manual Section 2.8.1)

The project tasks for the NIROP source area groundwater investigation are as follows.

- Investigation Support Tasks Section 14.1
- Field Tasks Section 14.2
- Analysis and QC Tasks Section 14.3
- Data Management and Records Tasks Section 14.4

The health and safety considerations for the work associated with this plan, including both potential physical and chemical hazards, are addressed in the Site-Specific Health and Safety Plan, to be submitted under separate cover.

14.1 Investigation Support Tasks

Logistical tasks required to support the field effort are described below.

14.1.1 Site Access and Mobilization/Demobilization

BAE is engaged in the development, delivery, and support of advanced defense, security and aerospace systems and many areas of the building require security clearance. Security requirements will be confirmed before initiating on-site work, but it is assumed that a birth certificate or confirmation of U.S. citizenship is required for all on-site personnel to work at the site.

Mobilization will consist of the delivery and secure storage of necessary equipment, materials, and supplies, along with the acquisition of personnel and vehicle access badges. The Resolution Consultants Field Operations Leader (FOL) or designee will coordinate with the Navy RPM to identify appropriate locations for the temporary storage of equipment and supplies. Fieldwork will be conducted in four separate phases as described below. After each phase, any equipment or IDW to remain on site will be secured. Final demobilization will consist of the prompt and timely removal of equipment, materials, and supplies from the site, at the completion of fieldwork. Demobilization also includes the cleanup and removal of waste generated during the investigation.

14.1.2 Utility Clearance

A minimum of two weeks prior to the commencement of any intrusive activities, Resolution Consultants will coordinate utility clearance with the property owner, the Navy, and the Gopher State One Call public utility locating service. A private utility locator may also be contracted



to locate private utilities at the site. The Resolution Consultants FOL will document the utility clearance process.

14.1.3 Coring Through Building Foundation

Concrete within the building is estimated to be up to approximately 1-foot thick. The concrete floor will be cut using a concrete coring drill by either the drilling subcontractor or other subcontractor prior to boring advancement.

14.2 Field Tasks and Quality Control

Field tasks will be overseen and documented on a daily basis. This will include maintenance of a daily log book(s) to document site conditions, on-site personnel, visitors to the site, work tasks completed, etc. At the conclusion of each work day, a diagonal line will be drawn across the unused portion of the log book page, signed, and dated to document the end of the days field activities.

14.2.1 Field Tasks

Tasks to be completed for the source area groundwater investigation are presented below.

- Completion of eight MIP borings continuously screened for VOCs and particle size from surface to approximately 80 feet bgs. Borings will be advanced commencing from downgradient to upgradient, to minimize potential cross-contamination resulting from VOCs liberated during penetration of finer-grained stratigraphic units.
- Completion of 27 vertical profile borings consisting of two separate side-by-side borings per location. The first boring consists of continuous direct push soil sampling with macro-core sampler to approximately 80 feet bgs to obtain stratigraphy. Vadose zone soil samples will be collected for VOC laboratory analysis every 5 feet from six vertical profile borings near the East Plating Room. The second side-by-side boring consists of groundwater sampling approximately every 5 feet using a retractable screen point sampler. All stratigraphic soil borings shall be completed prior to groundwater sampling in order to allow dissipation of any VOCs liberated during penetration of finer grained stratigraphic units.
- Completion of three vadose zone soil borings near the East Plating Room consisting of continuous soil sampling to the water table (approximately 20 feet bgs) with four analytical soil samples per boring.
- Flexibility within the field program will be achieved by reviewing the results of the MIP and vertical profile programs with the partnering team and obtaining consensus on the need for relocating or conducting additional explorations. This will include up to five vertical profile contingency borings.



- Installation and development of three monitoring wells. Nine soil samples will be collected from the three monitoring well soil borings (three samples per monitoring well boring) for VOC laboratory analysis. See Section 17.5.2 for information on well construction.
- Survey all MIP borings, vertical profile borings, and monitoring wells.
- Collection of groundwater samples at the three newly installed monitoring wells for analysis of VOCs, sulfate, nitrate, total and dissolved iron, methane, ethane, and ethene. In non-VOC sample containers approximately 10 percent of the sample container shall be unfilled to compensate for changes in pressure and temperature during shipping.
- Decontamination and site restoration.
- Waste handling.
- Completion of field documentation.

Sampling rationale, well construction details, and approach for the source area investigation is presented in Worksheet #17. Specific samples are listed in Worksheet #18.

At each vertical profile location, two side-by-side borings will be completed: the first boring to collect and log soil samples and the second boring to collect groundwater samples. To minimize the potential for the soil borings to affect the groundwater sample boring, to the extent practical, the soil borings will be:

- Located hydraulically downgradient from the groundwater borings;
- Conducted approximately two weeks before the groundwater borings at a given location; and
- Abandoned with high solids bentonite, without the use of concrete, immediately after completing the boring.

In the soil boring, soil samples will be collected continuously from the ground surface to the termination depth using Geoprobe macro-core soil sampler with new disposable liners. A geologist will record the lithology of the soil samples on the boring log sheet, including the borehole location; drilling information; and sampling information such as sample intervals, recovery, and sample description information. Samples from the soil core will also be carefully screened with a PID on approximately 1-foot intervals and the results recorded on the boring log sheet. For borings SB-102 through SB-104 and VP-4 through VP-9 near the East Plating Room, vadose zone soil samples will collected approximately every 5 feet for laboratory VOC analysis. The decision rule for sample



selection will be to select the segment of the core that reflects the highest level of impact for laboratory analysis. The primary criteria for sample selection will be visual NAPL observations, discoloration associated with reduced conditions, and PID screening. If the primary criteria do not suggest a specific zone with greater impact in the soil core, then secondary criteria, geologic observations (e.g., soil contacts, water table, etc.) will be used to identify the specific sample location. In addition, up to 10 additional soil samples will be collected from the vadose zone or beneath the water table for TOC and iron analysis, at the discretion of the field geologist. In these non-VOC sample containers approximately 10 percent of the sample container shall be unfilled to compensate for changes in pressure and temperature during shipping. The decision criteria for these samples is to obtain laboratory data from a range of soil types and depths. In the remaining borings, no vadose zone samples will be collected for laboratory analysis. During advancement and logging of the soil borings, the field geologist will note the thickness of fine grained silt and clay "confining" layers beneath the water table. For fine grained layers greater than 5 feet thick, the field geologist may collect a soil sample beneath the water table for VOC analysis since a groundwater sample cannot practically be collected from these fine grained units with a screen point sampler. Soil data collected beneath the water table will be used to help design potential remedial actions and clarify contaminant source areas.

Logging by the field geologist will be conducted as outlined in SOP 3-16 (Appendix B). The PID will be operated and calibrated as outlined in SOP 3-20 (Appendix B). Soil samples will be collected for laboratory analysis in accordance with procedures in SOP 3-21 (Appendix B).

For the second side-by-side boring at each location, up to 12 discrete groundwater samples will be collected from each vertical profile boring. One groundwater sample from each boring will be collected from approximately 7 feet below the observed water table and the remaining samples will be collected approximately every 5 feet. The remaining groundwater sampling locations will be selected to provide a vertical profile of the groundwater conditions in the source areas. As such, the decision criteria for groundwater sample collection will focus on areas of high MIP or PID response, areas above and below fine grained "confining" layers, and at the bottom of the boring. However, due to the short intervals between groundwater samples (5 feet) and the length of the sampling screen (3 feet), only minor adjustments to the groundwater sample depth will be made. The short sampling interval will enable a detailed assessment of the groundwater profile at each boring location. Groundwater samples will not be collected from fine grained units (e.g., silt and clay) due to time limitations and equipment limitations. Groundwater samples will be collected in accordance with the Geoprobe screen point sampler recommendations using a bladder pump (see Appendix D for Geoprobe screen point sampler SOP).



Groundwater sampling will be performed as follows.

- The boring will be advanced to the deepest groundwater sampling interval. The temporary well screen will then be exposed by retracting drill casing 3 feet (both Geoprobe SP 16 and SP 22 models of screen point samplers will be available for use during sampling and the most effective will be utilized).
- Clean, dedicated sample tubing and bladder pump will then be placed in the drill casing to the approximate mid-point of the temporary screen.
- The water will be pumped using a bladder pump until water clarity improves or 1 to 2 liters of water have been evacuated (field geologist will use professional judgment to determine when adequate purging has been completed).
- At the conclusion of purging, the sample will be collected directly into the pre-cleaned laboratory supplied volatile organic analysis vials. After sample collection, the container will be inspected for the presence of bubbles. If bubbles are observed, the sample shall be discarded and a new sample collected. If effervescence within the sample container continues to generate bubbles, the sample will be collected in an unpreserved sample container. The laboratory will be notified of this situation, as it will shorten the sample hold time.
- Upon completion of sampling, the tubing will be withdrawn and disposed. The bladder pump will be decontaminated and the drill casing, with exposed well screen, will be withdrawn to the next sample interval where sampling will again be conducted using the same method as outlined above.
- If the well screen is pulled through a thick fine-grained soil sequence (5 feet or thicker), the entire drill string will be withdrawn to the ground surface so the tooling, including the well screen can be decontaminated before re-advancing the tooling, with retractable well screen, to the desired sample depth, where the next sample will be collected.
- Note that groundwater samples will be collected approximately every 5 feet. However, no groundwater samples will be collected from fine grained (silt or clay) units of approximately 5-foot thickness or greater. In lieu of collecting groundwater samples from fine-grained units of 5 feet in thickness or greater, the field geologist will collect a soil sample from these units for VOC analysis during advancement of the first soil boring as previously described.

Based on USEPA and MPCA concerns, the Navy will consider using the "bottom down" sampling approach with decontamination of the screen point sampler between each sampling depth in the borings near the East Plating Room. This decision will be made by the field crew based upon the MIP responses, the thickness and frequency of fine grained soil units encountered, and the drilling conditions encountered / drilling depths achievable.



Equipment blanks will be performed at a rate of approximately 1 per 20 vertical profile groundwater samples (approximately 17 equipment blanks). Equipment blanks will be collected prior to initiation of a boring by pumping distilled water through the screen point sampler and bladder pump and tubing and placed directly into laboratory supplied containers. Sampling equipment will be decontaminated between boring locations as outlined in SOP 3-06 (Appendix B).

Groundwater samples and equipment blanks will be submitted to TriMatrix for laboratory analysis of VOCs by Method 8260B. Borings will be abandoned in accordance with MDH regulations immediately after advancement.

14.2.2 Quality Control Tasks – Field

The MIP will be calibrated daily, before use, in the middle of the day and at the end of day in accordance with the manufacturer's instructions. It is anticipated that two borings will be completed per day, so the mid-day calibration will be completed between boring locations. If calibration fails, results will need to be evaluated to determine which, if any, borings need to be redone.

All other field instruments will be calibrated daily, before use, and will be performed in accordance with the manufacturer's instructions.

14.3 Analytical Laboratory Tasks

Chemical analysis of groundwater will be performed by a subcontracted laboratory, TriMatrix. TriMatrix is MDH and DoD ELAP-accredited and their certificates are in Appendix C. Analyses will be performed in accordance with the analytical methods identified in Worksheets #23 and #30. TriMatrix will strive to meet the PALs shown in Worksheet #15 and will perform chemical analyses following laboratory-specific SOPs cited on Worksheet #23 and provided in Appendix C. The laboratory accreditation will be verified prior to the commencement of the field sampling effort to verify that the accreditations are current.

14.4 Data Management and Record Tasks

14.4.1 Documentation and Records

The PM will maintain project files which include, but are not limited to, project plans and specifications, maps and drawings, field forms, chain-of-custody (COC) records, laboratory analytical data packages, technical reports, correspondence, survey data, and other pertinent information. The project files will be maintained at the PM's office for the duration of the project. Laboratory data packages, survey data, reports and correspondence will also be maintained in NIRIS.



14.4.2 Analytical Data Packages

All analytical data packages for samples analyzed to either aid in defining the source area (VOCs), provide information on soil conditions (iron and TOC), or provide information on groundwater conditions (sulfate, nitrate, total and dissolved iron, methane, ethane and ethane) will be a legally defensible data package (typically referred to as USEPA Level IV). The package must allow complete reconstruction of the analytical test results to include but not limited to: results, units, percent moisture, dilutions, analytical batches, LOQs, LODs, summary QC information, laboratory control limits, initial and continuing calibration information, instrument chromatograms, and laboratory bench sheets.

Data packages for samples collected for IDW management and waste disposal will include summary QC information to include method blanks, surrogate, recoveries, and laboratory control sample (LCS) recoveries. Validation is not required for samples used for waste characterization and IDW management and the USEPA Level II data package is adequate.

14.4.3 Assessment/Audit Tasks

The FOL will review sample collection information, COC forms and field forms for compliance with this SAP on a daily basis. The analytical laboratory is accredited under DoD ELAP and is certified by the MDH.

14.4.4 Data Review Tasks

The analytical laboratory will verify that all data are complete for samples received.

All data will be verified by Resolution Consultants for compliance with the project specific MPC. Resolution Consultants will validate 10 percent of the VOC data at a level IV validation and the remaining 90 percent at a Level III as outlined in Worksheet #34-36. For each mobilization, the first 10 percent of the data submitted will undergo a Level IV data review. If there are no gross errors associated with the data, then the remaining data will undergo Level III validation. If gross errors are noted in the first 10 percent of the data validated, then Level IV data validation will continue until the issues have been resolved. Data defined as screening data on Worksheet #23 will undergo Level III validation only (i.e. results will not be recalculated). No validation will be performed for IDW samples used for disposal decisions. Validation personnel will be independent, and not employed by the laboratory analytical subcontractor. Validation will be conducted using general guidance in USEPA's *Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review* (2008) and *Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review* (2010) as applicable.



Analytical results will be validated before any decisions based on the data are made.

14.4.5 Data Tracking

Data are tracked from generation to archiving in the Resolution Consultants project-specific files. The Resolution Consultants PC (or designee) is responsible for tracking the samples collected and shipped to the laboratory. Upon receipt of the data packages from the laboratory, the Resolution Consultants PC will monitor the data validation effort, which includes verifying that the data packages are complete and results for all samples have been delivered by the laboratory.

14.4.6 Electronic Data

All electronic data will be compiled into a NIRIS Electronic Data Deliverable and loaded into the NIRIS database. The Resolution Consultants Data Manager maintains the electronic data files, and access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.



SAP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLES

(UFP-QAPP Manual Section 2.8.1)

15.1 Reference Limits and Evaluation Tables for Groundwater Samples

Matrix: Groundwater

Analytical Group: All

Analyte	CAS No.	Project Action Level (µg/L)	Project Action Level Source (µg/L)	Project Quantitation Limit Goal (µg/L)	Laboratory Limit of Quantitation ¹ (µg/L)	Laboratory Limit of Detection ¹ (µg/L)	Laboratory Detection Limit ¹ (µg/L)
VOCs:							
1,1,1-Trichloroethane	71-55-6	200	MCL	40	1	0.5	0.297
1,1-Dichloroethane	75-34-3	100	RAA	20	1	0.5	0.279
1,1-Dichloroethene	75-35-4	7	MCL	1.4	1	0.5	0.207
cis-1,2-Dichloroethene	156-59-2	50	HRL	10	1	0.5	0.202
Tetrachloroethene	127-18-4	5	HRL/MCL	1	1	0.5	0.16
trans-1,2-Dichloroethene	156-60-5	40	HRL	8	1	0.5	0.218
Trichloroethene	79-01-6	5	HRL/MCL	1	1	0.5	0.244
Vinyl chloride	75-01-4	0.2	HRL	0.04	1	0.5	0.135
Non-VOC Analytes:							
Sulfate	18785-72-3	--	--	2000	2000	1000	237
Nitrate	14797-55-8	10,000	HRL/MCL	100	100	50	12.6
Total Iron	7439-89-6	--	--	10	10	10	6.53
Dissolved Iron	7439-89-6	--	--	10	10	10	6.53
Methane	74-82-8	--	--	0.5	0.5	0.47	0.145
Ethane	74-84-0	--	--	1	1	0.91	0.242
Ethene	74-85-1	--	--	1	1	0.84	0.248

Notes:

¹ Laboratory quantitation, detection, and method detection limits, provided by TriMatrix Laboratories, are targets that are achievable under optimal conditions and may vary during the course of the project. Physical characteristics, such as matrix interference or dilutions due to high sample concentration, will affect the actual limits achieved.

Bolded/Shaded = Limits of quantitation and limits of detection exceed project action limit. The results will be used for decision making as long as values below the limits of quantitation are "J" qualified and discussed in the uncertainties section of the report.

- = no criteria listed
- HRL = health risk limit
- CAS = Chemical Abstract Service
- MCL = maximum contaminant level
- RAA = risk assessment advice
- µg/L = microgram per liter
- VOC = volatile organic compound



15.2 Reference Limits and Evaluation Tables for Soil Samples

Matrix: Soil

Analytical Group: All

Analyte	CAS No.	Project Action Level (mg/kg)	Project Action Level Source (mg/kg)	Project Quantitation Limit Goal (mg/kg)	Laboratory Limit of Quantitation ¹ (mg/kg)	Laboratory Limit of Detection ¹ (mg/kg)	Laboratory Detection Limit ¹ (mg/kg)
<i>VOCs:</i>							
1,1,1-Trichloroethane	71-55-6	472	SRV	94	0.05	0.025	0.0069
1,1-Dichloroethane	75-34-3	55	SRV	11	0.05	0.025	0.0109
1,1-Dichloroethene	75-35-4	60	SRV	12	0.05	0.025	0.0105
cis-1,2-Dichloroethene	154-59-2	22	SRV	4.4	0.05	0.05	0.0113
Tetrachloroethene	127-18-4	131	SRV	26	0.05	0.05	0.0138
trans-1,2-Dichloroethene	156-60-5	33	SRV	6.6	0.05	0.05	0.0116
Trichloroethene	79-01-6	46	SRV	9.2	0.05	0.025	0.0106
Vinyl chloride	75-01-4	2.2	SRV	0.44	0.05	0.025	0.0084
<i>Non-VOC Analytes:</i>							
Iron	7439-89-6	75,000	SRV	15000	5	1	0.619
Total organic carbon	7440-44-0	--	--	1,000	1,000	200	173

Notes:

¹ Laboratory quantitation, detection, and method detection limits, provided by TriMatrix Laboratories, are targets that are achievable under optimal conditions and may vary during the course of the project. Physical characteristics, such as matrix interference, moisture content, or dilutions due to high sample concentration, will affect the actual limits achieved.

-- + No criteria listed
 CAS = Chemical Abstract Service
 mg/kg = milligram per kilogram
 SRV = soil reference value
 VOC = Volatile organic compound



15.3 REFERENCE LIMITS AND EVALUATION TABLES FOR IDW SAMPLES

Matrix: Investigation-Derived Waste – Soil or Water

Analytical Group: All for contaminant extent

Analyte	CAS Number	Units	Project Action Level	Project Action Level Source	Project Quantitation Limit Goal	Laboratory Limit of Quantitation ¹	Laboratory Limit of Detection ¹	Laboratory Detection Limit ¹
TCLP Volatile Organic Compounds								
1,1-Dichloroethene	75-35-4	µg/L	700	CFR 261.24	70	1	0.5	0.207
1,2-Dichloroethane	107-06-2	µg/L	500	CFR 261.24	50	1	0.5	0.147
2-Butanone (MEK)	78-93-3	µg/L	200000	CFR 261.24	20000	50	2	1.62
Benzene	71-43-2	µg/L	500	CFR 261.24	50	1	1	0.319
Carbon tetrachloride	56-23-5	µg/L	500	CFR 261.24	50	1	1	0.229
Chlorobenzene	108-90-7	µg/L	100000	CFR 261.24	10000	1	0.5	0.214
Chloroform	67-66-3	µg/L	6000	CFR 261.24	600	1	1	0.255
Tetrachloroethene	127-18-4	µg/L	700	CFR 261.24	70	1	0.5	0.16
Trichloroethene	79-01-6	µg/L	500	CFR 261.24	50	1	1	0.244
Vinyl chloride	75-01-4	µg/L	200	CFR 261.24	20	1	0.5	0.135
TCLP Semivolatile Organic Compounds								
1,4-Dichlorobenzene	106-46-7	µg/L	7500	CFR 261.24	750	5	0.5	0.197
2,4,5-Trichlorophenol	95-95-4	µg/L	400000	CFR 261.24	40000	5	2.5	0.851
2,4,6-Trichlorophenol	88-06-2	µg/L	2000	CFR 261.24	200	5	2.5	0.992
2,4-dinitrotoluene	121-14-2	µg/L	130	CFR 261.24	13	5	1	0.475
2-Methylphenol	95-48-7	µg/L	200000	CFR 261.24	20000	5	2	0.475
4-Methylphenol	8001-28-3	µg/L	200000	CFR 261.24	20000	5	2	0.566
Hexachlorobenzene	118-74-1	µg/L	130	CFR 261.24	13	5	1	0.627
Hexachlorobutadiene	87-68-3	µg/L	500	CFR 261.24	50	5	1	0.395
Hexachloroethane	67-72-1	µg/L	3000	CFR 261.24	300	5	1	0.418
Nitrobenzene	98-95-3	µg/L	2000	CFR 261.24	200	5	2	0.585
Pentachlorophenol	87-86-5	µg/L	100000	CFR 261.24	10000	5	5	1.26
Pyridine	110-86-1	µg/L	5000	CFR 261.24	500	50	10	2.51
TCLP Metals								
Arsenic	7440-38-2	µg/L	5000	CFR 261.24	500	500	100	35
Barium	7440-39-3	µg/L	100000	CFR 261.24	10000	350	10	2.71
Cadmium	7440-43-9	µg/L	1000	CFR 261.24	100	50	10	3.52
Chromium	7440-47-3	µg/L	5000	CFR 261.24	500	250	20	4.62
Lead	7439-92-1	µg/L	5000	CFR 261.24	500	250	50	14.6
Mercury	7439-97-6	µg/L	200	CFR 261.24	20	0.2	0.1	55.1
Selenium	7782-49-2	µg/L	1000	CFR 261.24	500	200	100	32.6
Silver	7440-22-4	µg/L	5000	CFR 261.24	500	50	10	4.08



Analyte	CAS Number	Units	Project Action Level	Project Action Level Source	Project Quantitation Limit Goal	Laboratory Limit of Quantitation ¹	Laboratory Limit of Detection ¹	Laboratory Detection Limit ¹
TCLP Pesticides								
Chlordane (Technical)	12789-03-6	µg/L	30	CFR 261.24	25	25	25	7.1
Endrin	72-20-8	µg/L	20	CFR 261.24	10	5	5	1.6
Heptachlor	76-44-8	µg/L	8	CFR 261.24	5	5	5	1.35
Heptachlor epoxide	1024-57-3	µg/L	8	CFR 261.24	5	5	5	1.6
Lindane (gamma-BHC)	58-89-9	µg/L	400	CFR 261.24	40	5	5	1.65
Methoxychlor	72-43-5	µg/L	10000	CFR 261.24	1000	5	1	0.25
Toxaphene	8001-35-2	µg/L	500	CFR 261.24	250	100	62.5	38.4
Others								
PCBs (Soil)	1336-36-3	mg/kg	8	SRV	1	0.33	0.013	0.0069
PCB-1016	12674-11-2	mg/kg	8	SRV	1	0.33	0.013	0.0017
PCB-1221	11104-28-2	mg/kg	8	SRV	1	0.33	0.013	0.0021
PCB-1232	11141-16-5	mg/kg	8	SRV	1	0.33	0.013	0.0021
PCB-1242	53469-21-9	mg/kg	8	SRV	1	0.33	0.013	0.0069
PCB-1248	12672-29-6	mg/kg	8	SRV	1	0.33	0.013	0.0025
PCB-1254	11097-69-1	mg/kg	8	SRV	1	0.33	0.013	0.0034
PCB-1260	11096-82-5	mg/kg	8	SRV	1	0.33	0.013	0.0028
PCBs (Water)	1336-36-3	µg/L	0.5	MCL	0.5	0.2	0.08	0.0426
PCB-1016	12674-11-2	µg/L	0.5	MCL	0.5	0.2	0.08	0.0299
PCB-1221	11104-28-2	µg/L	0.5	MCL	0.5	0.2	0.08	0.0336
PCB-1232	11141-16-5	µg/L	0.5	MCL	0.5	0.2	0.08	0.0426
PCB-1242	53469-21-9	µg/L	0.5	MCL	0.5	0.2	0.08	0.0274
PCB-1248	12672-29-6	µg/L	0.5	MCL	0.5	0.2	0.08	0.0211
PCB-1254	11097-69-1	µg/L	0.5	MCL	0.5	0.2	0.08	0.0138
PCB-1260	11096-82-5	µg/L	0.5	MCL	0.5	0.2	0.08	0.0234
pH (Soil)	NA	pH Units	>2 and ≤12.5	CFR 261	0.1	0.1	NA	NA
pH Water) [§]	NA	pH Units	>2 and ≤12.5	CFR 261	0.1	0.1	NA	NA
Paint Filter Test	NA	milliliter	-	-	-	1	NA	NA
Ignitability (Soil)	NA	°F	NA	N/A	68	68	NA	NA
Flashpoint (Water)	NA	°F	60	CFR 261	68	68	NA	NA

Notes:

¹ Laboratory quantitation, detection, and method detection limits, provided by TriMatrix Laboratories, are targets that are achievable under optimal conditions and may vary during the course of the project. Physical characteristics, such as matrix interference, moisture content, or dilutions due to high sample concentration, will affect the actual limits achieved.

CAS = Chemical Abstract Service
 CFR = Code of Federal Regulations
 °F = degree Fahrenheit
 MCL = maximum contaminant level
 mg/kg = milligrams per kilogram

NA = not applicable
 PCB = polychlorinated biphenyl.
 SRV = Soil Reference Value
 TCLP = toxicity characteristic leaching procedure
 µg/L = micrograms per liter



SAP WORKSHEET #16: PROJECT/TIMELINE TABLE
 (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		
Prepare/Submit Internal Draft UFP SAP to Navy (draft revised after January 2013 meeting)	Resolution Consultants	10/11/12	3/12/13	Internal Draft UFP SAP	3/12/13
Navy Internal Review of UFP SAP Complete	Navy	3/12/13	4/19/13	"Response to Navy Comments"	NA
Prepare/Submit Draft UFP SAP to Navy, USEPA and MPCA	Resolution Consultants	3/12/13	3/15/13	Draft SAP	3/15/13
Regulatory Review of Draft UFP SAP	USEPA, MPCA	3/15/13	4/25/13	NA	NA
Prepare/Submit response to comments	Resolution Consultants	4/25/13	5/24/13	"Response to USEPA/MPCA Comments"	5/24/13
Regulatory review of response to comments	USEPA, MPCA	5/24/13	5/28/13	Conference Call to Discuss	5/28/13
Submit final response to comments UFP SAP	Resolution Consultants	5/28/13	6/19/13	Final Response to Comments	6/19/13
Submit final UFP SAP	Resolution Consultants	6/19/13	7/1/13	Final UFP SAP	7/1/13
Fieldwork: Eight MIP borings	Resolution Consultants	7/15/2013	7/19/13	MIP Logs will be sent to Navy, MPCA and USEPA	7/22/13
Fieldwork: 27 vertical profile borings and 3 shallow vadose zone borings	Resolution Consultants	8/5/13	9/13/13	Boring Logs	10/1/13
Laboratory results	TriMatrix Laboratories	7/21/13	10/4/13	Laboratory data packages and electronic data deliverables	10/4/13
Data validation	Resolution Consultants	8/15/13	10/25/13	Chemical Data Quality Review Report	10/25/2013
Review MIP results and soil and groundwater analytical results with partnering team. Determine if any additional borings are needed to fill perceived data gaps. Determine monitoring well locations.	Navy, USEPA, MPCA	10/7/13	10/18/13	Consensus on Locations for any additional borings to fill data gaps. Determine Monitoring Well Locations based on MIP and Groundwater Lab Data	NA
Fieldwork: Complete any contingency borings to fill data gaps identified by partnering team.	Resolution Consultants	10/28/13	10/30/13	NA	NA



Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Fieldwork: Install 3 monitoring wells	Resolution Consultants	11/18/13	11/22/13	NA	NA
Fieldwork: Sample 3 monitoring wells	Resolution Consultants	12/6/13	12/6/13	NA	NA
Laboratory results from well installation and sampling	TriMatrix	11/22/13	12/27/13	Laboratory data packages and electronic data deliverables	12/27/13
Data validation	Resolution Consultants	1/2/14	1/24/14	Chemical Data Quality Review Report	1/24/14
Prepare/Submit Internal Draft Source Area Investigation Report to Navy	Resolution Consultants	9/23/14	2/28/14	Internal Draft Source Area Investigation Report	2/28/14
Navy Review of Internal Draft Source Area Investigation Report	Navy	2/28/14	3/14/14	NA	NA
Prepare Response to Navy Comments and Revised Draft Source Area Investigation Report	Resolution Consultants	3/14/14	3/28/14	"Response to Navy Comments" and Revised Internal Draft Source Area Investigation Report	3/28/14
Navy Review and Approval of "Response to Comments" and Revised Draft Source Area Investigation Report	Navy	3/28/14	4/11/14	NA	NA
Prepare/Submit Draft Source Area Investigation Report to Navy, USEPA and MPCA	Resolution Consultants	4/11/14	4/18/14	Draft Source Area Investigation Report	4/18/14
Regulatory Review of Draft Source Area Investigation Report	USEPA, MPCA	4/18/14	5/18/14	NA	NA
Prepare/Submit "Response to Comments" and Final Draft Source Area Investigation Report	Resolution Consultants	5/18/14	6/18/14	"Response to USEPA/MPCA Comments" and Final Draft Source Area Investigation Report	6/18/14
Regulatory Review of "Response to Comments" and Final Draft Source Area Investigation Report	USEPA, MPCA	6/18/14	7/18/14	NA	NA
Prepare/Submit Final Source Area Investigation Report	Resolution Consultants	7/18/14	8/1/14	Final Source Area Investigation Report	8/1/14

Notes:

MPCA = Minnesota Pollution Control Agency
 MIP = membrane interface probe
 NA = Not applicable

UFP SAP = Uniform Federal Policy Sampling and Analysis Plan
 USEPA = United States Environmental Protection Agency



SAP WORKSHEET #17: SAMPLING DESIGN AND RATIONALE

(UFP-QAPP Manual Section 3.1.1)

17.1 Sampling Approach

As described in Worksheet #10, the investigation is being conducted to define the nature and contaminant concentrations within the subsurface soil and groundwater at three potential source areas beneath the NIROP building, including shallow vadose zone soils near the East Plating Room. The sampling locations will be biased toward the suspected source areas and downgradient flow paths identified during previous soil and groundwater investigations at the site (refer to Source Investigation Map Methodology, February 2012, for summary/interpretation of historic data). Figure 10-1 illustrates the proposed boring locations.

A phased approach will be employed with information obtained in initial phases used to optimize sample locations and well placement in subsequent phases. The sample locations are biased based on known site characteristics. The phased approach will allow step-out sampling to more specifically delineate source areas and address data gaps, as needed. The rationale for sample numbers, locations, depths, media type, and analyses are summarized below. Details on sample objectives, locations, and depths are also included on Figure 1 and Worksheet #18. Since QC samples are collected on a regular basis as a percentage of lab analytical samples, they are outlined in Worksheet #20.

Investigation activities will be conducted over four to five separate mobilizations. The proposed timing for these mobilizations was presented in Worksheet #16. These mobilizations are as follows:

1. Mobilization 1 includes advancement of eight MIP borings. Results of the MIP borings will be interpreted to evaluate the nature of the potential source area at the East Plating Room.
2. Mobilization 2 includes advancement of 27 vertical profile borings and three additional shallow vadose zone soil borings. Preliminary results/recommendations will be submitted to the Project Team for discussion.
3. Mobilization 3 includes installation of any contingency vertical profile borings to fill data gaps identified by the project team after review of the initial data (assume up to five borings to address data gaps).
4. Mobilization 4 includes installation of three permanent monitoring wells using sonic drilling methods with soil sampling and well development.
5. Mobilization 5 includes sampling of the three new monitoring wells. Soil borings and monitoring wells will surveyed by a Minnesota Professional Land Surveyor.



This worksheet presents the design and rationale of the sampling and analysis program to be conducted during the Source Area Investigation. The sampling approach, based on professional judgment, is biased to investigate areas most likely to contain "worst-case" concentrations. Specifically, the sampling plan was developed using information on assumptions regarding potential contaminant distribution and data generated during previous investigations. A summary table, including sample identification numbers, depth, relevant SOP, and applicable laboratory and field analyses, is included as Worksheet #18. SOPs for field activities are summarized in Worksheet #21 and included as Appendix B. Professional judgment may be used to adjust sampling locations and/or depths in the field. Examples of criteria which may lead to changes to the sampling plan include refusal, drill rig access issues, utility locations, initial MIP responses, etc.

Samples collected during this investigation will be submitted to TriMatrix for chemical analyses. Analytical methods are identified in Worksheet #23. Laboratory SOPs are listed in Worksheet #23 and are available upon request. The total numbers of sample analyses to be performed for each target analyte or analytical group are identified in Worksheets #18 and #20. Worksheets #19 and #30 present a summary of the sample analyses, container types and volumes, preservation requirements, and holding times.

Planned field QC samples will include field duplicates and equipment rinsate blanks (for non-dedicated sampling equipment). Worksheet #12 presents the field QC sample summary. Additional sample volume will be collected as necessary for laboratory QC analysis of MS/MSD samples.

17.2 First mobilization - MIP Boring Installation,

The rationale for the eight MIP borings is to provide adequate vertical and horizontal MIP data to support overall remedial planning and to supplement the vertical profile program.

MIP boring locations will be laid out in the field using paint. The concrete will be cut using a concrete coring drill by a subcontractor. The MIP borings will be advanced by Vironex, Inc. using a direct push drilling rig. For safety purposes, the first 5 feet of the boring may be advanced with a hand auger prior to drilling with the Geoprobe direct push rig. MIP soil profiling equipment will be supplied and operated in the field by Vironex, Inc. The Geoprobe MIP SOP from the manufacturer is included in Appendix D. During advancement, MIP borings will be logged continuously from ground surface to the termination depth of each boring for VOCs and EC. The total depth of each boring is expected to be approximately 80 feet bgs; however, borings may need to be advanced deeper in some locations in order to encounter the targeted confining layer. Drilling to these depths will reach the practical limits of the drilling equipment and refusal may occur prior to reaching the lower confining layer. Encountering the lower confining layer will be determined



based on EC readings collected during boring advancement. A log of the MIP and EC results will be produced for each boring location.

Borings will be abandoned in accordance with MDH regulations immediately after advancement. The MIP equipment will be decontaminated between boring locations. If refusal occurs during boring advancement, the boring location will be off-set slightly and another attempt will be made. If refusal occurs a second time, no additional attempts at that boring location will be made. If refusal becomes routine while advancing the MIP, then the field team will demobilize and an alternative drilling method will be selected. The field team will then remobilize to the field and complete the eight MIP borings, as needed.

A summary of MIP boring location rationale is listed below.

- MIP-1 will be advanced immediately adjacent to the north side of the East Plating Room building to determine soil and groundwater conditions in that area.
- MIP-2 through MIP-5 are located along the southern edge of the East Plating Room to investigate soil and groundwater impacts in this area.
- MIP-6 through MIP-8 will form a transect approximately 100 feet further south of the East Plating Room to investigate downgradient soil and groundwater impacts in this area.

17.3 Second Mobilization - Vertical Profile Borings and Shallow Vadose Zone Borings

Up to 27 vertical profile borings and three shallow vadose zone soil borings will be advanced with a direct push rig. Three shallow vadose zone borings will be drilled to the water table only (approximately 20 feet bgs) and the vertical profile borings will be installed to approximately 80 feet bgs. Six of the vertical profile borings near the East Plating Room (VP-4 through VP-9) will also be used for shallow vadose zone sampling. Eight of the vertical profile borings will be installed adjacent to previously installed MIP borings and two will be installed near the groundwater extraction wells. Below is a summary of boring location rationale:

- Borings VP-1 through VP-3 are located near or downgradient of AOC-17 to provide an additional vertical profile data on the magnitude of TCE impacts in the vicinity of AOC-17 and to determine if there is a connection with impacts found in MS-31. Three borings with a total of 36 groundwater samples for VOC analysis will be conducted.
- Borings VP-4 through VP-9 and SB-102 through SB-104 are located around the East Plating Room to investigate soil and groundwater TCE impacts adjacent to the East Plating Room. Four borings to 20 feet and six borings to 80 feet bgs will be conducted with 72



groundwater samples for VOC analysis, and 36 soil samples for VOC analyses will be conducted.

- Borings VP-10 through VP-12 will form a transect approximately 100 feet further south of the East Plating Room to investigate downgradient groundwater impacts to depths of 80 feet bgs in this area. Three borings with a total of 36 groundwater samples for VOC analyses will be conducted.
- Borings VP-13 and VP-14 will be advanced adjacent to and downgradient of boring P09 (installed during the RI for OU3) to determine current groundwater TCE impacts to depths of 80 feet bgs in this area. Two borings with a total of 24 groundwater samples for VOC analyses will be conducted.
- Borings VP-15 and VP-16 are located between the East Plating Room and the extraction wells to assess TCE distribution in soil and groundwater TCE to depths of 80 feet bgs. Two borings with a total of 24 groundwater samples for VOC analyses will be conducted.
- Borings VP-17 and VP-18 are located near well MS-33 to assess groundwater TCE impacts to depths of 80 feet bgs in the 7th and Broadway area where deep impacts in groundwater have been detected. Two borings with a total of 24 groundwater samples for VOC analyses will be conducted.
- Borings VP-19 and VP-20 are located near wells UD-67S/68I/69D to assess groundwater TCE impacts near and downgradient of the wells to depths of 80 feet bgs. Two borings with a total of 24 groundwater samples for VOC analyses will be conducted.
- Boring VP-21 is located upgradient of MS-33 and UD-67S/68I/69D to assess groundwater TCE impacts and evaluate if there is a connection to the Paint Shop at depths up to 80 feet bgs. One boring with a total of 12 groundwater samples for VOC analyses will be conducted.
- Borings VP-22 through VP-27 are located between the extraction wells and the suspected source areas to assess TCE distribution in soil and groundwater at depths up to 80 feet bgs. Six borings with a total of 72 groundwater samples for VOC analyses will be conducted.

The primary purpose of the borings is to determine vadose zone soil conditions near the East Plating Room, characterize subsurface soil stratigraphy, provide data to help determine monitoring well locations and screen depths, provide analytical data to be used in evaluating remedial options, and to better understand the distribution of TCE in the groundwater in the source areas and downgradient areas.



Equipment blanks will be performed at a rate of approximately 1 per 20 vertical profile groundwater samples (approximately 17 equipment blanks). Equipment blanks will be collected prior to initiation of a boring.

17.4 Third Mobilization – Contingency Vertical Profile Borings

The MIP results and preliminary vertical profile boring laboratory analytical results will be presented to the partnering team to determine if there are data gaps. If data gaps are identified, additional vertical profile boring locations will be completed during the second mobilization, as agreed upon by the partnering team. It is assumed that up to five contingency vertical profile borings will be sufficient to address data gaps. The vertical profile contingency borings will be completed using the same methodology as outlined in Section 17.3.

17.5 Fourth Mobilization - Monitoring Well Installation and Soil Sampling

Three groundwater monitoring wells will be installed in the intermediate groundwater zone during the third mobilization. The purpose of the wells is to provide baseline TCE concentrations in the groundwater prior to completing any pilot test work (e.g., EZVI injections) and to provide points to monitor the effectiveness of the pilot test. Well locations will be selected based on the MIP and vertical profile groundwater sample results, most likely focusing on the source area exhibiting the highest TCE concentrations/greatest extent and downgradient areas. The results of the MIP, vertical profile, and contingency borings will be reviewed and used to identify the horizontal and vertical position of the long term monitoring wells. The proposed vertical and horizontal locations of the wells will then be presented to the project team for their concurrence prior to installation of the wells.

17.5.1 Soil Sample Collection from Monitoring Well Borings

Monitoring well borings will be installed using a sonic drilling rig. The borings will be advanced using sonic drilling techniques with soil cores collected continuously from the ground surface to the terminus of the boring (anticipated to be up to 80 feet bgs). Boreholes will be 4-inch diameter core tube with 6-inch diameter override casing. The boreholes will be screened and logged in a manner identical to that outlined in Section 14.2.1.

Nine soil samples will be collected from the three monitoring well soil borings (three samples per monitoring well boring) for laboratory analysis in accordance with procedures in SOP 3-21 (Appendix B). Soil sample locations and depths will be selected to obtain samples representative of the various soil types present. The decision criteria outlined in Section 14.2.1 will also be used for sample selection. Soil samples will be submitted to TriMatrix for laboratory analysis of VOCs by Method 8260B, iron by Method SW6010C, and TOC by the Walkley-Black Procedure. VOC results from soil samples will be used to assess the extent of source area VOC impacts. TOC and iron



results from soil samples will be used to provide data for evaluating remedial options and to establish baseline site concentrations.

17.5.2 Monitoring Well Installation

Monitoring wells will be installed using a sonic drill rig and will be constructed in accordance with applicable MDH regulations and the procedures outlined in SOP 3-12 (Appendix B). Monitoring well construction will be similar to the existing site monitoring wells and will use 2-inch diameter National Sanitation Foundation approved stainless steel well screen and carbon steel riser pipe. Well screens will be 5 feet in length and will have a 0.010-inch slot size. Caps will be fitted to the bottom of each well screen. Pipe sections and bottom caps will be flush-jointed.

The filter packs will consist of 20/30 clean silica sand installed using a tremie pipe from the base of screen extending to 2 feet above the top of the screen. A 2-foot thick seal of bentonite chips will be installed above the sand layer. After the bentonite chips are hydrated, the annulus above the bentonite seal will be filled with high-solids bentonite grout using a tremie pipe.

Wells will be completed at the surface with a 12-inch diameter flush mount protective steel casing capable of withstanding heavy traffic. The flush mount covers will be set in a new 5-foot by 5-foot by 1-foot thick concrete pad, tied into the existing cement floor, with a 2-inch rise in the center tapering to grade at the edges. A layer of fine sand will be installed above the grout slurry and inside the flush mount box. The tops of well risers will be set approximately 6 inches below grade. Lockable gripper caps will be installed on well riser tops. Monitoring well installations will comply with applicable MDH regulations. A variance will be requested as needed.

17.5.3 Monitoring Well Development

Following installation, the three monitoring wells will be developed to improve hydraulic communication with the surrounding aquifer and to evacuate fine-grained sediments which may have accumulated within the well during installation. Monitoring wells will generally be developed in accordance with procedures in SOP No. 3-13 (Appendix B) using the surge and purge method. Well development will not commence until approximately 24 hours after well installation. It is anticipated that the wells will be developed using mechanical surging and a submersible pump. Development will continue until turbidity has stabilized (less than 50 nephelometric turbidity units, if possible). Special care will be taken to develop the wells properly in order to ensure adequate hydraulic connection between the monitoring well and the aquifer.

17.6 Fifth Mobilization - Groundwater Sampling

Groundwater samples will be collected from each of the three monitoring wells during the fourth mobilization. Groundwater elevations will also be concurrently collected from selected nearby



monitoring wells to be determined at the time of sampling. Groundwater samples will be collected to obtain a baseline of groundwater conditions and to aid in the evaluation of remedial options. Groundwater samples may also be collected from the wells to monitor future pilot study effectiveness.

Groundwater sampling will be completed approximately two weeks after well development. Groundwater samples will be collected using a bladder pump and low-flow sampling procedures described in SOP 3-14 (Appendix B). Prior to sampling, the monitoring wells will be gauged for depth to water and to check for the presence of light and dense NAPLs with an interface probe. To confirm that groundwater samples are representative of the formation being investigated, field measurements of water level/drawdown, temperature, pH, specific conductance, ORP, DO, and turbidity will be collected during well purging. Purging will be considered complete when three consecutive readings of the parameters listed above reach less than 10 percent variability. The sampling pump and other non-dedicated equipment that comes into contact with the purge or sample water will be decontaminated between monitoring well locations as outlined in SOP 3-06 (Appendix B). Sample tubing and other dedicated equipment (bailers, drop clothes, etc) will be discarded between each sample.

One equipment blank will be collected for the groundwater samples by pumping distilled water through the bladder pump and tubing; and placed directly into laboratory supplied containers. Groundwater samples from the new wells and the equipment blank will be submitted to TriMatrix for analysis of VOCs by Method 8260B, sulfate and nitrate by Method 9056A, total and dissolved iron by Method 6010C, and dissolved gasses (methane, ethane, ethane) by Method RSK 175.



SAP WORKSHEET #18: LOCATION-SPECIFIC SAMPLING METHODS/SOP REQUIREMENTS TABLE

(UFP-QAPP Manual Section 3.1.1)

Sample Location	Sample Identification	Matrix	Estimated Depth (feet bgs)	Objective	Analytical Group	Number of Samples	Sampling Standard Operating Procedure Reference (Appendix B and D)
SB-102	SB102-S-XX	Soil	4 samples above WT	6 (see notes)	VOCs	4	SOP 3-21
SB-103	SB103-S-XX	Soil	4 samples above WT	6	VOCs	4	SOP 3-21
SB-104	SB104-S-XX	Soil	4 samples above WT	6	VOCs	4	SOP 3-21
VP-1	VP1-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	2	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-2	VP2-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	2	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-3	VP3-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	2	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-4	VP4-S-XX	Soil	4 samples above WT	6	VOCs	4	SOP 3-21
	VP4-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-5	VP5-S-XX	Soil	4 samples above WT	6	VOCs	4	SOP 3-21
	VP5-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-6	VP6-S-XX	Soil	4 samples above WT	6	VOCs	4	SOP 3-21
	VP6-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-7	VP7-S-XX	Soil	4 samples above WT	6	VOCs	4	SOP 3-21
	VP7-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-8	VP8-S-XX	Soil	4 samples above WT	6	VOCs	4	SOP 3-21
	VP8-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-9	VP9-S-XX	Soil	4 samples above WT	6	VOCs	4	SOP 3-21
	VP9-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14



Sample Location	Sample Identification	Matrix	Estimated Depth (feet bgs)	Objective	Analytical Group	Number of Samples	Sampling Standard Operating Procedure Reference (Appendix B and D)
VP-10	VP10-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-11	VP11-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-12	VP12-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-13	VP13-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	5 and investigation of P09 area	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-14	VP14-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	5 and investigation of P09 area	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-15	VP15-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1, 5	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-16	VP16-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	1, 5	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-17	VP17-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	3	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-18	VP18-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	3	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-19	VP19-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	4	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-20	VP20-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	4	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-21	VP21-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	4	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-22	VP22-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	5	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-23	VP23-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	5	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-24	VP24-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	5	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-25	VP25-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	5	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-26	VP26-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	5	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
VP-27	VP27-W-XX	Groundwater	12 depths ⁽¹⁾ , TBD	5	VOCs	12	Geoprobe Screen Point System SOP & SOP 3-14
MS-57I	MS57I-S-XX	Soil	TBD	8	VOCs, iron, TOC	3	SOP 3-21



Sample Location	Sample Identification	Matrix	Estimated Depth (feet bgs)	Objective	Analytical Group	Number of Samples	Sampling Standard Operating Procedure Reference (Appendix B and D)
	MS57I-Date	Groundwater	TBD	1, 5 and 7	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane, ethene	1	SOP 3-14
MS-58I	MS58I-S-XX	Soil	TBD	8	VOCs, iron, TOC	3	SOP 3-21
	MS58I-Date	Groundwater	TBD	1, 5 and 7	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane, ethene	1	SOP 3-14
MS-59I	MS59I-S-XX	Soil	TBD	8	VOCs, iron, TOC	2	SOP 3-21
	MS59I-Date	Groundwater	TBD	1, 5 and 7	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane, ethene	1	SOP 3-14
Quality Assurance, Quality Control, and IDW Samples							
SB-YYY	FDZZ	Soil (Duplicate)	sample above WT	9	VOCs	2	SOP 3-21
VP-YY	FDZZ	Soil (Duplicate)	sample above WT	9	VOCs	4	SOP 3-21
MS-YYI	FDZZ	Soil (Duplicate)	TBD	9	VOCs, iron, TOC	1	SOP 3-21
SB-YYY	SBYYY-S-XX (MS will be indicated in COC notes)	Soil (Matrix Spike)	TBD ⁽³⁾	9	VOCs	1	SOP 3-21
SB-YYY	SBYYY-S-XX (MSD will be indicated in COC notes)	Soil (Matrix Spike Duplicate)	TBD ⁽³⁾	9	VOCs	1	SOP 3-21
VP-YY	VPYY-S-XX (MS will be indicated in COC notes)	Soil (Matrix Spike)	TBD ⁽³⁾	9	VOCs	1	SOP 3-21
VP-YY	VPYY-S-XX (MSD will be indicated in COC notes)	Soil (Matrix Spike Duplicate)	TBD ⁽³⁾	9	VOCs	1	SOP 3-21
MS-YYI	MSYYI-S-XX (MS will be indicated in COC notes)	Soil (Matrix Spike)	TBD ⁽³⁾	9	VOCs, iron, TOC	1	SOP 3-21
MS-YYI	MSYYI-S-XX (MSD will be indicated in COC notes)	Soil (Matrix Spike Duplicate)	TBD ⁽³⁾	9	VOCs, iron, TOC	1	SOP 3-21
VP-YY	FDZZ	Groundwater (Duplicate)	7 feet below water table	9	VOCs	33	Geoprobe Screen Point System SOP & SOP 3-14



Sample Location	Sample Identification	Matrix	Estimated Depth (feet bgs)	Objective	Analytical Group	Number of Samples	Sampling Standard Operating Procedure Reference (Appendix B and D)
MS-YYI	FDZZ	Groundwater (Duplicate)	TBD	9	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane, ethene	1	SOP 3-14
VP-YY	VPYY-XX (MS will be indicated in COC notes)	Groundwater (Matrix Spike)	TBD ⁽³⁾	9	VOCs	17	Geoprobe Screen Point System SOP and SOP 3-14
VP-YY	VPYY-XX (MSD will be indicated in COC notes)	Groundwater (Matrix Spike Duplicate)	TBD ⁽³⁾	9	VOCs	17	Geoprobe Screen Point System SOP and SOP 3-14
MS-YYI	MSYYI-Date (MS will be indicated in COC notes)	Groundwater (Matrix Spike)	TBD	9	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane, ethene	1	SOP 3-14
MS-YYI	MSYYI-Date (MSD will be indicated in COC notes)	Groundwater (Matrix Spike Duplicate)	TBD	9	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane, ethene	1	SOP 3-14
VP-YY	VPYY-EB01	VP (Equipment Blank)	NA	9	VOCs	17	See Worksheet #17
WGEB01	WGEB01	Monitoring well (Equipment Blank)	NA	9	VOCs, sulfate, nitrate, total and dissolved iron, methane/ethane, ethene	1	See Worksheet #17
IDW-Liquid	IDW-Liquid	Waste	NA	10	TCLP VOCs, TCLP SVOCs, TCLP Metals, TCLP pesticides, PCBs, Ignitability, pH ⁽²⁾	1	SOP 3-05
IDW-Soil	IDW-Soil	Waste	NA	10	TCLP VOCs, TCLP SVOCs, TCLP Metals, TCLP pesticides, PCBs, Ignitability, pH, paint filter test ⁽²⁾	1	SOP 3-05



Notes:

- ⁽¹⁾ = One depth will be at 7 feet below the water tables, other depths to be determined based on membrane interface probe (MIP) and soil lithology. Some soil samples may be collected in lieu of groundwater samples where fine grained confining layers are encountered beneath the water table. Up to 10 soil samples will be also analyzed for TOC and iron from locations selected by the field geologist.
- ⁽²⁾ = Test to be performed as required by the disposal firm.
- ⁽³⁾ = Samples expected to have relatively lower VOC concentrations based on the MIP boring results will be selected for MS/MSDs.
- bgs = Below ground surface
COC = chain of custody
IDW = investigative derived waste
MS/MSD = matrix spike/matrix spike duplicate
NA = Not Applicable
PCB = polychlorinated biphenyl
S = soil sample
SOP = Standard Operating Procedure
SVOC = semivolatle organic compound
TBD = to be determined
TCLP = toxicity characteristic leaching procedure
TOC = total organic carbon
VOCs = volatile organic compounds
VP = vertical profiling boring
W = Water sample
WT = water table
XX = sample depth, to be determined
YY = sample location identification
ZZ = Field duplicates to be numbered sequentially and association with parent sample documented in the log book to maintain blind duplicates.

Objectives for each exploration location:

1. Determine the concentrations and vertical/horizontal concentrations of VOCs in the presumed East Plating Room source area and downgradient areas,
2. Determine the magnitude of concentrations and vertical profile of impacts in the presumed AOC-17 source area and downgradient areas,
3. Determine if there is a connection between the elevated concentrations detected at monitoring well MS-33I and the East Plating Room presumed source area and determine vertical concentrations at 7th and Broadway area,
4. Evaluate groundwater concentrations downgradient from the Paint Shop and determine if they extend onto the Naval Industrial Reserve Ordnance Plant site toward the extraction wells and evaluate vertical concentrations near Wells UD-67S / 68I / 69D,
5. Assess groundwater concentrations of trichloroethene (TCE) and associated degradation products downgradient from source areas,
6. Evaluate the vadose zone concentrations of TCE and associated degradation products in shallow soil at the East Plating Room,
7. Estimate the baseline groundwater concentrations of total and dissolved iron, sulfate, nitrate, methane, ethane, and ethene in groundwater near the source areas, and
8. Determine the general soil types and baseline concentrations of total organic carbon, iron, and TCE along with its associated degradation products for remedial planning.
9. quality assurance/quality control samples
10. IDW samples.



SAP WORKSHEET #19: ANALYTICAL METHODS/SOP REQUIREMENTS TABLE

(UFP-QAPP Manual Section 3.1.1)

Laboratory Name and Address: TriMatrix Laboratories, 5560 Corporate Exchange Court, SE, Grand Rapids, Michigan 49512

Laboratory Point of Contact/Project Chemist: Walt Roudebush, roudebushw@trimatrixlabs.com (616) 975-4561

Matrix	Analytical Group	Analytical and Preparation Method/ TriMatrix SOP Reference ⁽⁴⁾	Containers (number, size, and type)	Sample Volume	Preservation Requirements ⁽¹⁾	Maximum Holding Time ⁽¹⁾ (preparation/analysis)
<i>Site Characterization Samples:</i>						
Groundwater	VOCs	SW-846 5030/8260B GR-03-105/GR-04-104	(3) 40 mL glass volatile vials	40 mL	Hydrochloric acid to a pH less than 2; Cool to 0-6°C; no headspace ⁽²⁾	14 days (7 days if sample is not preserved with hydrochloric acid)
Groundwater	Sulfate/Nitrate	SW-846 9056A GR-02-113	1 liter glass or plastic	1 liter	Cool to 0-6°C	Nitrate – 48 hours Sulfate – 28 days
Groundwater	Metals - Total Iron	SW-846 3010A/6010C GR-01-147/GR-01-100	500 mL PTFE container with a screw cap	500 mL	pH<2 with HNO ₃ , Cool to 0-6°C	180 days
Groundwater	Metals - Dissolved Iron	SW-846 3010A/6010C GR-01-147/GR-01-100	500 mL PTFE container with a screw cap	500 mL	Field filter pH<2 with HNO ₃ , Cool to 0-6°C	180 days
Groundwater	Methane, ethane, ethene	RSK 175 GR-03-130	(3) 40 mL glass volatile vials	40 mL	Hydrochloric acid to a pH less than 2; Cool to 0-6°C; no headspace ⁽²⁾	14 days
Soil	VOCs	SW-846 5035/8260B GR-04-105/GR-04-104	(3) 40 mL glass volatile vial plus 2 oz HDPE container	5 grams	1 vial with 5 ml Methanol; Cool to 0-6°C 2 vials with 5 ml sodium bisulfate ⁽⁵⁾	14 days
Soil	Metals - Iron	SW-846 3050B/6010C GR-01-137/GR-01-100	(1) 4 oz HDPE container	1.25 grams	None	180 days
Soil	TOC	Walkley-Black GR-06-105	(1) 4 oz glass jar	100 grams	Cool to 0-6°C	28 days



Laboratory Name and Address: TriMatrix Laboratories, 5560 Corporate Exchange Court, SE, Grand Rapids, Michigan 49512
Laboratory Point of Contact/Project Chemist: Walt Roudebush, roudebushw@trimatrixlabs.com (616) 975-4561

Matrix	Analytical Group	Analytical and Preparation Method/ TriMatrix SOP Reference ⁽⁴⁾	Containers (number, size, and type)	Sample Volume	Preservation Requirements ⁽¹⁾	Maximum Holding Time ⁽¹⁾ (preparation/analysis)
<i>Investigative Derived Waste Samples⁽³⁾:</i>						
IDW Soil	TCLP VOCs	SW1311/8260B GR-01-119/GR-04-104	(1) 4 oz glass jar	50 grams	Minimal headspace in sample container, Cool to 0-6°C	Samples leached within 14 days; leachate analyzed within 14 days of leachate generation
	TCLP SVOCs, TCLP pesticides	SW1311/SW8270C and SW8081B GR-01-119/GR-04-103/GR-03-120	(1) 8 oz glass jar	125 grams	Cool to 0-6°C	Samples leached within 14 days; leachate extracted within 7 days of leachate generation and extracts analyzed within 40 days following extraction
	TCLP Metals	SW1311/SW6010C/SW7470A GR-01-119/GR-01-100/GR-01-128				Samples leached within 180 days (28 days for mercury); leachate digested and analyzed within 180 days (28 days for mercury)
	PCBs	SW8082A GR-03-128	(1) 8 oz glass jar	50 grams	Cool to 0-6°C	None
	Ignitability	SW1020A GR-18-124	(1) 8 oz HDPE container	5 grams	Cool to 0-6°C	28 days
	pH	SW9045D GR-07-113		50 grams	Cool to 0-6°C	7 days
	Paint filter test	9095B GR-19-102		100 grams	Cool to 0-6°C	28 days



Laboratory Name and Address: TriMatrix Laboratories, 5560 Corporate Exchange Court, SE, Grand Rapids, Michigan 49512
Laboratory Point of Contact/Project Chemist: Walt Roudebush, roudebushw@trimatrixlabs.com (616) 975-4561

Matrix	Analytical Group	Analytical and Preparation Method/ TriMatrix SOP Reference ⁽⁴⁾	Containers (number, size, and type)	Sample Volume	Preservation Requirements ⁽¹⁾	Maximum Holding Time ⁽¹⁾ (preparation/analysis)
IDW Liquid	VOCs	SW8260B GR-03-105/GR-04-104	(3) unpreserved 40 mL Field Operations Leader VOA vials	40 mL	Cool to 0-6°C	7 days (assuming the liquid IDW is its own leachate)
	SVOCs pesticides	SW8270C and SW8081B GR-04-103/GR-03-120	(4) 1 liter glass	2 liter	Cool to 0-6°C	Extracted within 7 days and extracts analyzed within 40 days following extraction (assuming the liquid IDW is its own leachate)
	Metals	SW6010C/7470A GR-01-100/ GR-01-123	(1) 500 mL HDPE container	100 mL	Cool to 0-6°C	180 days, 28 days for mercury (assuming the liquid IDW is its own leachate)
	PCBs	SW8082A GR-03-128	(2) 1 liter glass	1 liter	Cool to 0-6°C	None
	Ignitability, pH	SW1020A, 9040C GR-18-124/GR-07-100	(1) 500 mL HDPE container	200 mL	Cool to 0-6°C	28 days (Ignitability) and As Soon As Possible (pH) (assuming the liquid IDW is its own leachate)

Notes:

- (1) = Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted
 - (2) = Table 4-1 of SW846 recommends that samples analyzed for vinyl chloride and styrene be analyzed as unpreserved. Vinyl chloride is not the primary VOC of interest and a second analysis with an unpreserved vial is not proposed.
 - (3) = Samples collected to determine disposal options only. Bottle requirements are provided to aid samplers.
 - (4) = TriMatrix SOPs provided in Appendix C.
 - (5) = Sodium bisulfate preserved samples will be collected for vadose zone soil samples only. Samples preserved in methanol to be analyzed, sodium bisulfate preserved samples will only be analyzed if reporting limits cannot be achieved with methanol preserved sample.
- | | | | |
|------|-------------------------------|------|---------------------------------|
| °C | = degrees Celsius | SOP | = Standard Operating Procedure |
| HDPE | = high-density polyethylene | SVOC | = semivolatile organic compound |
| IDW | = investigation-derived waste | TOC | = total organic carbon |
| mL | = milliliter | VOA | = volatile organic analysis |
| oz | = ounce | VOCs | = volatile organic compound |
| PCB | = polychlorinated biphenyl | | |
| PTFE | = polytetrafluoroethylene | | |



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 (10% frequency)	No. of MS/MSDs ⁽¹⁾ (5% frequency)	No. of Equip. Blanks (5% frequency)	No. of Trip Blanks ⁽²⁾	Total No. of Samples to Lab
Groundwater from Vertical Profile Borings (27 borings, up to 12 sample depths each)	VOCs	324	33	17	17	20	394
Soil from Vertical Profile Borings, Vadose Zone Borings and Monitoring Well Borings	VOCs	45	7	3	0	5	57
	Iron	19	1	1	0	0	20
	TOC	19	1	1	0	0	20
Groundwater from Monitoring Wells	VOCs	3	1	1	1	1	6
	Sulfate/Nitrate	3	1	1	1	0	5
	Total Iron	3	1	1	1	0	5
	Dissolved Iron	3	1	1	1	0	5
	Methane/Ethane/Ethene	3	1	1	1	0	5

Note:

¹ Although matrix spike/matrix spike duplicate (MS/MSD) samples are not typically considered field quality control samples, they are included here because location determination is often established in the field. MS/MSD samples are not included in the total number of samples sent to the laboratory.

² One per cooler containing samples for volatile organic compound (VOC) analysis.

TOC = total organic carbon



SAP WORKSHEET #21: PROJECT SAMPLING SOP REFERENCES TABLE

(UFP-QAPP Manual Section 3.1.2)

Field SOPs Reference Table

SOP Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Yes/No)	Comments
SOP-3-01	Utility Clearance, Revision 0; June 2012	Resolution Consultants	Toning Equipment	No	Appendix B
SOP-3-02	Log Books, Revision 0, May 2012	Resolution Consultants	None	No	Appendix B
SOP-3-03A	Record Keeping, Sampling Labeling, and Chain-of-Custody, Revision 0, May 2012	Resolution Consultants	None	No	Appendix B
SOP-3-04A	Sample Handling, Storage, and Shipping, Revision 0, May 2012	Resolution Consultants	None	No	Appendix B
SOP-3-05	Investigation-Derived Waste Management, Revision 0, May 2012	Resolution Consultants	None	No	Appendix B
SOP-3-06	Equipment Decontamination, Revision 0, May 2012	Resolution Consultants	Interface Probe, Pumps, Drill Tooling, Surge Block	No	Appendix B
SOP-3-12	Monitoring Well Installation, Revision 0, May 2012	Resolution Consultants	Sonic Drill Rig	No	Appendix B
SOP-3-13	Monitoring Well Development, Revision 0, June 2012	Resolution Consultants	Pumps and Surge Block	No	Appendix B
SOP-3-14	Monitoring Well Sampling, Revision 0, May 2012	Resolution Consultants	Interface Probe, Bladder Pump, Water Quality Meter, Turbidity Meter	No	Appendix B
SOP-3-16	Soil and Rock Classification, Revision 0, August 2012	Resolution Consultants	None	No	Appendix B
SOP-3-19	Headspace Screening for VOCs, Revision 0, May 2012	Resolution Consultants	Photoionization Detector	No	Appendix B
SOP-3-20	Operation and Calibration of a Photoionization Detector, Revision 0, May 2012	Resolution Consultants	Photoionization Detector	No	Appendix B
SOP-3-21	Surface and Subsurface Soil Sampling, Revision 0, May 2012	Resolution Consultants	Terra Core® or Cut Off Syringe, Scale	No	Appendix B



Field SOPs Reference Table

SOP Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Yes/No)	Comments
SOP-3-24	Water Quality Parameter Testing, Revision 0, May 2012	Resolution Consultants	Water Quality Meter, Turbidity Meter	No	Appendix B
Technical Bulletin No. MK3010	Geoprobe Membrane Interface Probe SOP, May 2003, Revised April, 2012	Geoprobe Systems, Inc.	Membrane Interface Probe	No	Appendix D
Technical Bulletin No. MK3142	Geoprobe ® Screen Point 16 Groundwater Sampler, November 2006	Geoprobe Systems, Inc.	Screen Point 16 Groundwater Sampler	No	Appendix D
Technical Bulletin No. MK3173	Geoprobe ® Screen Point 22 Groundwater Sampler, April, 2010	Geoprobe Systems, Inc.	Screen Point 22 Groundwater Sampler	No	Appendix D

Notes:

If contradictions are noted between the Sampling and Analysis Plan (SAP) and the SOP, the requirements of the SAP will take precedence.

SOP = Standard Operating Procedure

VOCs = volatile organic compounds



SAP WORKSHEET #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment Calibration, Maintenance, Testing, and Inspection Table							
Field Equipment	Activity	Frequency	Acceptance Criterion	Corrective Action	Responsible Person	SOP Reference	Comments
YSI 556 Water Quality Meter	Visual Inspection Calibration/Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or replacement	Resolution Consultants FOL or designee	Manufacturer's Guidance Manual, SOP-3-24	To be used to determine purge completion
Turbidity Meter (LaMotte 2020 or equivalent)	Visual Inspection Calibration/Verification	Daily Beginning and end of day	Manufacturer's guidance Calibrations must bracket expected values. Initial Calibration Verification must be <5 Nephelometric Turbidity Unit.	Operator correction or replacement	Resolution Consultants FOL or designee	Manufacturer's Guidance Manual, SOP-3-24	To be used to determine purge completion
Photoionization Detector (MiniRAE 3000 or equivalent)	Visual Inspection Calibration/Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or replacement	Resolution Consultants FOL or designee	Manufacturer's Guidance Manual, SOP-3-20	To be used to assist with determination of sampling depths and for safety monitoring
Water Level Indicator and Oil/Water Interface Probe	Visual Inspection and Field checks as per manufacturer	Daily	0.01 foot accuracy	Operator correction or replacement	Resolution Consultants FOL or designee	Manufacturer's Guidance Manual, SOP-3-24	None
Membrane Interface Probe	Calibration/Verification	Before and After each Boring	Manufacturer's guidance	Operator correction or replacement	Vironex, Inc. Operator	Geoprobe Technical Bulletin No. MK3010	None

Notes:

- FOL = Field Operations Leader
- SOP = Standard Operating Procedure



SAP WORKSHEET #23: ANALYTICAL SOP REFERENCES TABLE

(UFP-QAPP Manual Section 3.2.1)

Laboratory Name and Address: TriMatrix Laboratories, Inc. 5560 Corporate Exchange Court, SE, Grand Rapids, Michigan 49512

Laboratory Point of Contact/Project Chemist: Walt Roudebush, roudebushw@trimatrixlabs.com (616) 975-4561

TriMatrix SOP Number ¹	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM	Modified for Project Work? (Y/N)
GR-03-105	VOCs, 2/28/12, Revision 6.3	Definitive	Aqueous: VOCs	NA	No	N
GR-04-105	Closed System Purge and Trap and Extraction for VOCs, 8/25/12, Revision 1.4	Definitive	Soil: VOCs	NA	No	N
GR-04-104	VOCs by Purge and Trap Capillary Column GC/MS, 1/25/12, Revision 4.7	Definitive	Aqueous and Soil: VOCs	GC/MS	No	N
GR-01-147	Block Digestion of Total Metals in Water for ICP Method 6010C, 8/30/12, 0.5	Screening	Aqueous: Metals	NA	No	N
GR-01-137	Block Digestion of Solids for ICP and ICPMS	Screening	Soil: Metals	NA	No	N
GR-01-100	ICP Atomic Emission Spectroscopy-Perkin Elmer OPTIMA-3300DV/5300DV, 7/22/11, 5.9	Screening	Aqueous and Soil: Metals	ICP-AES	No	N
GR-06-105	Organic Carbon, Walkley-Black	Screening	Soil: Total organic carbon	NA	No	N
GR-02-113	Determination of Inorganic Anions by Ion Chromatography, 8/3/12, 3.0	Screening	Water: Sulfate, Nitrate	Ion Chromatograph	No	N
GR-03-130	Dissolved Methane, Ethane, and Ethene in Water by Headspace Equilibrium and Gas Chromatography, 9/9/11, 0.5	Screening	Water: methane, ethane, ethene	GC	No	N
GR-01-119	TCLP, 7/23/12, 2.4	Screening	Aqueous and Soil IDW: TCLP	None	No	N
GR-01-123	Mercury by Semi-Automated Cold Vapor Atomic Absorption, 9/9/11, Revision 5.8	Screening	Aqueous and Soil IDW: mercury	Cold vapor atomic absorption	No	N



Laboratory Name and Address: TriMatrix Laboratories, Inc. 5560 Corporate Exchange Court, SE, Grand Rapids, Michigan 49512

Laboratory Point of Contact/Project Chemist: Walt Roudebush, roudebushw@trimatrixlabs.com (616) 975-4561

TriMatrix SOP Number ¹	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM	Modified for Project Work? (Y/N)
GR-03-120	Organochlorine Pesticides Analysis by Gas Chromatography, 9/9/11, 4.6	Screening	Aqueous and Soil IDW: Pesticides	GC with electron capture detector	No	N
GR-03-128	PCBs by Gas Chromatography, 2/17/12, Revision 2.7	Screening	Aqueous and Soil IDW: PCBs	GC with electron capture detector	No	N
GR-04-103	Base/Neutral/Acid Compounds by GC /MS	Screening	Aqueous and Soil IDW: SVOCs	GC/MS	No	N
GR-07-100	Potentiometric pH, 1/12/12, 3.5	Screening	Aqueous IDW: pH	pH Meter	No	N
GR-07-113	Potentiometric pH in Soil and Waste, 1/12/12, Revision 0.4	Screening	Soil IDW: pH	pH Meter	No	N
GR-18-124	Setaflash Closed-Cup Flashpoint, 4/4/13, 1.5	Screening	Aqueous and soil IDW: Ignitability	Koehler Rapid Tester Model K16502, Closed-cup	No	N
GR-19-102	Paint Filter Liquids Test, 8/3/12, 1.2	Screening	Soil IDW: free liquids	NA	No	N

Notes:

- ¹ = Laboratory SOPs provided in Appendix C
- GC/MS = gas chromatograph/mass spectrometer
- ICP = inductively coupled plasma spectroscopy
- ICP-AES = atomic emission spectroscopy
- ICPMS = inductively coupled plasma mass spectrometry
- IDW = investigation-derived waste
- NA = not available
- PCB = polychlorinated biphenyl
- QSM = Quality Systems Manual
- SOP = Standard Operating Procedure
- SVOC = semivolatile organic compound
- TCLP = Toxicity Characteristic Leaching Procedures
- VOC = volatile organic compound
- Y/N = yes/no



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	TriMatrix SOP Reference
GC/MS	Tuning	Prior to ICAL and at the beginning of each 12-hour period	<u>Refer to method for specific ion criteria.</u>	Retune instrument and verify. Rerun affected samples.	Analyst, Supervisor	TriMatrix SOP GR-04-104 GR-04-103
GC/MS	Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation $\leq 20\%$ for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Analyst, Supervisor	TriMatrix SOP GR-04-104 GR-04-103
GC/MS	CCV	Daily, before sample analysis and every 12 hours of analysis time	<ol style="list-style-type: none"> Average RF for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050 %D/drift for all target compounds and surrogates: VOCs and SVOCs $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). 	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Supervisor	TriMatrix SOP GR-04-104 GR-04-103



Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	TriMatrix SOP Reference
GC/MS	ICAL Minimum five-point ICAL for all analytes	ICAL prior to sample analysis	<p>1. <u>Average response factor for SPCCs:</u> VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane.</p> <p><u>SVOCs ≥ 0.050.</u></p> <p>2. RSD for RFs for CCCs: VOCs and SVOCs $\leq 30\%$ and one option below:</p> <p>Option 1: RSD for each analyte $\leq 15\%$.</p> <p>Option 2: linear least squares regression $r \geq 0.995$.</p> <p>Option 3: non-linear regression COD $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).</p>	Correct problem then repeat ICAL.	Analyst, Supervisor	TriMatrix SOP GR-04-104 GR-04-103
GC/MS	ICV	Once after each ICAL	All analytes within $\pm 20\%$ of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Supervisor	TriMatrix SOP GR-04-104 GR-04-103
GC/MS	RRT Evaluation	With each sample	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Supervisor	TriMatrix SOP GR-04-104 GR-04-103



Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	TriMatrix SOP Reference
GC/FID	ICAL for all analytes	Minimum five point ICAL for all project analytes prior to sample analysis and if CCV criteria have not been met	<p>One of the options below:</p> <p>Option 1: RSD for each analyte < 20%.</p> <p>Option 2: linear least squares regression ≥ 0.995.</p> <p>Option 3: non-linear regression: COD $r^2 > 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).</p>	Correct problem, then repeat ICAL.	Analyst, Supervisor	TriMatrix SOP GR-03-130
GC/FID	Retention time window position establishment for each analyte	Once per ICAL and at the beginning of the analytical shift	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	Analyst, Supervisor	TriMatrix SOP
GC/FID	Second SCV	Immediately following ICAL	All project analytes within + 20% of expected value from the ICAL.	Correct problem, rerun second source standard. If that fails, repeat ICAL.	Analyst, Supervisor	GR-03-130
GC/FID	CCV	Prior to sample analysis, after each 10 field samples, and at the end of the analytical sequence	All project analytes within established retention time windows. All project analytes within + 20% of expected value from the ICAL.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Supervisor	TriMatrix SOP
GC/ECD	Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation $\leq 15\%$ for both DDT and Endrin.	Correct problem then repeat breakdown check.	Analyst, Supervisor	TriMatrix SOPs: GR-03-120 GR-03-128
GC/ECD	Minimum five-point ICAL for all analytes	ICAL prior to sample analysis	<p>One of the options below:</p> <p>Option 1: RSD for each analyte $\leq 20\%$.</p> <p>Option 2: linear least squares regression: $r \geq 0.995$.</p> <p>Option 3: non-linear regression: COD $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).</p>	Correct problem then repeat ICAL	Analyst, Supervisor	TriMatrix SOPs: GR-03-120 GR-03-128



Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	TriMatrix SOP Reference
GC/ECD	Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	Analyst, Supervisor	TriMatrix SOPs: GR-03-120 GR-03-128
GC/ECD	Second source calibration verification (ICV)	Immediately following ICAL	All project analytes within established retention time windows. All project analytes within $\pm 20\%$ of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Supervisor	TriMatrix SOPs: GR-03-120 GR-03-128
GC/ECD	CCV	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence	All project analytes within established retention time windows. All project analytes within $\pm 20\%$ of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Supervisor	TriMatrix SOPs: GR-03-120 GR-03-128
ICP CVAA	ICAL ICP: minimum one high standard and a calibration blank CVAA: minimum 5 standards and a calibration blank	Daily ICAL prior to sample analysis	If more than one calibration standard is used, $r^2 \geq 0.995$.	Correct problem, then repeat ICAL.	Analyst, Supervisor	TriMatrix SOP GR-01-100
ICP CVAA	Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	All analytes within $\pm 10\%$ of expected value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Supervisor	TriMatrix SOP GR-01-100
ICP CVAA	CCV	CCV after every 10 field samples and at the end of the analytical sequence	ICP: All analytes within $\pm 10\%$ of expected value. CVAA: within $\pm 20\%$ of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Supervisor	TriMatrix SOP GR-01-100
ICP (ICP Only)	Low-level calibration check standard	Daily, after one-point ICAL	Within $\pm 20\%$ of true value.	Correct problem, then reanalyze.	Analyst, Supervisor	TriMatrix SOP GR-01-100



Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	TriMatrix SOP Reference
ICP (ICP Only)	ICS	At the beginning of an analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes <LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within $\pm 20\%$ of true value	Terminate analysis; locate and correct problem, reanalyze ICS, reanalyze all samples.	Analyst, Supervisor	TriMatrix SOP GR-01-100
ICP CVAA	Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analyte detected > LOD	Correct problem. Re-prepare and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst, Supervisor	TriMatrix SOP GR-01-100
ICP (ICP Only)	Linear Dynamic range/High level Check	Every 6 months and with major maintenance	Within $\pm 10\%$ of true value.	NA	Analyst, Supervisor	TriMatrix SOP GR-01-100
Ion Chromatograph	ICAL for all analytes (minimum three standards and one calibration blank)	ICAL prior to sample analysis	$r \geq 0.995$	Correct problem, then repeat ICAL.	Analyst, Supervisor	TriMatrix SOP GR-02-113
Ion Chromatograph	ICV (second source)	Once after each ICAL, prior to beginning a sample run	All analytes within $\pm 10\%$ of true value and retention times within appropriate windows.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Supervisor	TriMatrix SOP GR-02-113
Ion Chromatograph	Midrange CCV	After every 10 field samples and at the end of the analysis sequence	All project analytes within established retention time windows. Within $\pm 10\%$ of true value.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Supervisor	TriMatrix SOP GR-02-113



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Notes:

%D	=	percent difference
%RSD	=	relative standard deviation
CCC	=	calibration check compounds
CCV	=	continuing calibration verification
CV	=	calibration verification
COD	=	Coefficient of the determination
CVAA	=	cold vapor atomic absorption
DDT	=	dichlorodiphenyltrichloroethane
ECD	=	electron capture detector
FID	=	flame ionization detector
GC/MS	=	gas chromatograph/mass spectrometer
ICAL	=	initial calibration
ICP/MS	=	inductively coupled plasma/spectroscopy
ICS	=	interference check solutions
ICV	=	initial calibration verification
LOD	=	limit of detection
QC	=	quality control
r ²	=	correlation coefficient
RRT	=	relative retention times
RST	=	relative standard deviation
SCV	=	Source Calibration Verification
SOP	=	Standard Operating Procedure
SPCC	=	Spill Prevention, Control, and Countermeasure
SVOC	=	semivolatile organic compound
TriMatrix	=	TriMatrix Laboratories
VOCs	=	Volatile organic compounds



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	TriMatrix SOP Reference
GC/MS	Check for leaks, replace gas line filters, recondition or replace trap, replace column, clean injection port/liner	VOCs SVOCs	Monitor instrument performance via Continuing Calibration Verification	As needed	No maintenance is required as long as instrument QC meets DoD QSM criteria	Replace connections, clean source, replace gas line filters, replace trap, replace GC column, clip column, replace injection port liner, clean injection port, replace Electron Multiplier	Analyst, Supervisor	TriMatrix SOP GR-04-104
GC/FID	Replace septa, replace inlet liner, clip column, clean or replace FID jet, bake out detector, recondition column.	Methane, Ethane, Ethene	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	TriMatrix SOP GR-03-130
GC/ECD	Replace septa, replace inlet liner, clip column, bake out detectors, recondition column.	Pesticides 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/noise is elevated.	Per instrument manufacturer's instructions.	Inspect system, correct problem, perform new initial calibration, and reanalyze 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	TriMatrix SOP Reference
ICP	Change pump tubing, clean nebulizer, change torch, clean sample cone/skimmer cone	Metals	Monitor instrument performance via Continuing Calibration Verification and Blank	As needed	No maintenance is required as long as instrument QC meets DoD QSM criteria	Change pump tubing, change torch and nebulizer, clean cone; recalibrate and reanalyze affected data	Analyst, Supervisor	TriMatrix SOP GR-01-100
CVAA	Flush all tubing, replace pump tubing, replace Perma-pure drying system membrane or entire assembly, replace activated charcoal in exhaust line.	Mercury	Check for discoloration in Perma-pure drying system.	Flush tubing daily. Change tubing at least weekly. Check Perma-pure system for discoloration monthly. Replace Perma-pure inner membrane every six months. Replace charcoal annually.	Per instrument manufacturer's instructions.	Inspect system and correct problem.	Metals Analyst	TriMatrix SOP GR-01-123
Ion Chromatograph	Check and change inline sample filter as necessary	Sulfate, Nitrate	Check to make sure conductivity <1.0, check eluent lines for air bubbles, check suppressor for leaks	Perform checks daily, do maintenance as necessary	Conductivity <1.0	Inspect system and correct problem per manufacturer's instructions; Perform new initial calibration.	Wet Chemistry Analyst	GR-02-113

Notes:

- CVAA = Cold vapor atomic absorption
- DoD = Department of Defense
- ECD = electron capture detector
- FID = flame ionization detector
- GC/MS = gas chromatograph/mass spectrometer
- ICP = Inductively coupled plasma spectroscopy
- PCB = polychlorinated biphenyl
- QC = quality control
- QSM = Quality Systems Manual
- SOP = Standard Operating Procedure
- SVOC = semivolatile organic compound
- TriMatrix = TriMatrix Laboratories
- VOCs = volatile organic compounds



SAP WORKSHEET #26: SAMPLE HANDLING SYSTEM

(UFP-QAPP Manual Appendix A)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT	
Sample Collection (Personnel/Organization):	Field Operations Leader/Resolution Consultants
Sample Packaging (Personnel/Organization):	Field Operations Leader/Resolution Consultants
Coordination of Shipment (Personnel/Organization):	Field Operations Leader/Resolution Consultants
Type of Shipment/Carrier:	Overnight via FedEx or UPS
SAMPLE RECEIPT AND ANALYSIS	
Sample Receipt (Personnel/Organization):	Sample Custodian / TriMatrix Laboratories
Sample Custody and Storage (Personnel/Organization):	Sample Custodian / TriMatrix Laboratories
Sample Preparation (Personnel/Organization):	Laboratory Chemists / TriMatrix Laboratories
Sample Determinative Analysis (Personnel/Organization):	Laboratory Chemists / TriMatrix Laboratories
SAMPLE ARCHIVING	
Field Sample Storage (Number of days from sample collection):	60 Days
Sample Extract/Digestate Storage (Number of days from extraction/digestion):	60 Days
SAMPLE DISPOSAL	
Personnel/Organization:	Sample Custodian / TriMatrix Laboratories
Number of Days from Analysis:	60 Days



SAP WORKSHEET #27: SAMPLE CUSTODY REQUIREMENTS

(UFP-QAPP Manual Section 3.3.3)

27.1 Sample Nomenclature, Sample Collection Documentation, Handling, and Tracking Procedures

The following sections outline the procedures that will be used to document project activities and sample collection, handling, tracking, and custody procedures during the investigation. All forms must be filled in as completely as possible.

27.1.1 Sample Nomenclature

Sample labeling will be conducted in general accordance with the procedures outlined in Worksheet 18. Nomenclature for soil samples includes the soil boring identification number, sample media (S=soil, W=water), sample depth, and sample type code (i.e., EB=equipment blank). Nomenclature for aqueous samples from borings includes the boring identification number, sample media, sample depth, and sample type code. Nomenclature for aqueous samples from monitoring wells includes the monitoring well identification number, date, and sample type code. Trip blanks will be labeled sequentially (i.e., TB01, TB02, TB03). Samples to be used for MS/MSD will be labeled MS/MSD on the container label and noted on the COC; however, "MS/MSD" will not be part of the unique sample identifier in order to maintain consistency with the project database.

Duplicates will be collected as blind duplicates, labeled sequentially (i.e., FD01, FD02) and no time of collection will be indicated on the COC form. A record of the duplicate pairs will be maintained in the field logbook.

Worksheet #18 provides anticipated sample identifiers for this scope of work.

27.1.2 Sample Collection Documentation

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, and monitoring well construction logs. Field logbooks utilized on this project will consist of a bound, water-resistant logbook. All pages of the logbook will be numbered sequentially and observations will be recorded with indelible ink. Alternatively, boring logs may be recorded electronically using a tablet computer (e.g., EDGE software and Trimble Yuma rugged tablet computer).

Field sample log sheets will be used to document sample collection details and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration.



For sampling and field activities, the following types of information will be recorded in the field logbook as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Activities involved with the sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made on site
- Visitor names, affiliations, arrival and departure times
- Health and safety issues, including personal protective equipment

27.1.3 Sample Handling and Tracking System

Resolution Consultants personnel will collect the samples. Samples will be preserved as appropriate based on the analytical method. The laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 0 to 6 degrees Celsius (°C) until delivery to the laboratory. Samples will be placed in appropriate containers, packaged by Resolution Consultants' personnel and placed on ice in coolers under COC. All coolers will contain a temperature blank. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottles during shipment. The cooler will be secured using strapping tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis.

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., FedEx, UPS). For all samples collected, COC forms will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the COC form. Once received by the laboratory, receipt will be documented on the COC form and the samples will be checked in. The samples will remain under COC throughout the analysis period to ensure their integrity is preserved. Further details on COC procedures are provided in SOP 3-03 (Appendix B).

27.2 Field Sample Custody Procedures

COC protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with SOP 3-04.

A sample is under custody if:

- The sample is in the physical possession of an authorized person.
- The sample is in view of an authorized person after being in his/her possession.
- The sample is placed in a secure area by an authorized person after being in his/her possession.
- The sample is in a secure area, restricted to authorized personnel only.

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the form each time a sample transfer occurs. Integrity of the samples collected will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report.

The Resolution Consultants FOL is responsible for the care and custody of the samples collected until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the COC form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs. Laboratory SOPs are available upon request.

27.3 Laboratory Chain-of-Custody

Coolers are received and checked for proper temperature. A sample cooler receipt form will be filled out to note conditions and any discrepancies. The COC form will be checked against the sample containers for accuracy. Samples will be logged into the laboratory Information Management System and given a unique log number which can be tracked through processing.



The laboratory PC will notify the Resolution Consultants FOL verbally or via e-mail of any problems on the same day that an issue is identified.



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE

(UFP-QAPP Manual Section 3.4)

Matrix		Groundwater, Soil				
Analytical Group		VOCs				
Analytical Method		SW-846 8260B				
SOP Reference		TriMatrix SOP GR-04-104				
QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory 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. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	Correct problem; reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	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. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
Surrogates	All field and QC samples	QC acceptance criteria specified in DoD QSM Version 4.2	Reanalyze if sufficient sample is available. If reanalysis confirms failing recoveries, report and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
LCS	One LCS per preparatory batch	QC acceptance criteria specified in DoD QSM Version 4.2	Reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
Internal Standards	In all field samples and standards	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard	Inspect MS or GC for malfunctions. Reanalyze all samples with internal standard failures. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	Retention time ± 30 seconds; EICP area within -50% to +100% of midpoint of ICAL
MS/MSD	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.

Notes:

DoD QSM = Department of Defense Quality Systems Manual
 EICP = Extracted ion current profile
 GC = gas chromatograph
 ICAL = initial calibration
 LCS = laboratory control sample
 LOQ = limit of quantitation
 MS = Mass Spectrometer

MS/MSD = matrix spike/matrix spike duplicate
 QA = quality assurance
 QC = quality assurance
 RPD = relative percent difference
 SOP = Standard Operating Procedure
 VOC = volatile organic compound



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix		Groundwater, Soil				
Analytical Group		Iron				
Analytical Method		SW-846 6010C				
SOP Reference		TriMatrix SOP GR-01-100				
QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ 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. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	Correct problem; re-prepare and/or reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > ½ 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. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
LCS	One LCS per preparatory batch	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
Sample Duplicate or MSD	One per preparatory batch	RPD ≤ 20%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Precision	RPD ≤ 20%
Matrix Spike	One per preparatory batch per matrix	QC acceptance criteria specified in DoD QSM Version 4.2	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
Dilution Test	One per preparatory batch	Five-fold dilution must agree within ± 10% of the original measurement for samples with concentrations > 50 x LOQ	Perform Post Digestion Spike	Analyst, Supervisor, QA Manager	Accuracy Bias	Five-fold dilution must agree within ± 10% of the original measurement for samples with concentrations > 50 times LOQ



Matrix		Groundwater, Soil				
Analytical Group		Iron				
Analytical Method		SW-846 6010C				
SOP Reference		TriMatrix SOP GR-01-100				
QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Post Digestion Spike	When dilution test fails, the analyte concentration in all samples < 50 x LOD, or the MS/MSD recoveries fail.	Recovery 80-120%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 80-120%

Notes:

DoD QSM = Department of Defense Quality Systems Manual
 LCS = laboratory control sample
 LOD = limit of detection
 LOQ = limit of quantitation

MSD = matrix spike duplicate
 QA = quality assurance
 QC = quality assurance
 RPD = relative percent difference
 SOP = Standard Operating Procedure



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*Sampling and Analysis Plan — Source Area Investigation
Naval Industrial Reserve Ordnance Plant, Fridley Minnesota
SAP Worksheet #28
Revision: 0; July 2013*

SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix		Soil				
Analytical Group		Total Organic Carbon				
Analytical Method		Walkley-Black				
SOP Reference		TriMatrix SOP GR-06-105				
QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > LOD	Correct problem; reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > LOD
LCS	One LCS per preparatory batch	Recovery 85-115%	Reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 85-115%
Laboratory Duplicate	One per preparatory batch	RPD 20%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	RPD 20%

Notes:

LCS = laboratory control sample
QA = quality assurance
LOD = limit of detection

QC = quality assurance
RPD = relative percent difference
SOP = Standard Operating Procedure



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix		Groundwater				
Analytical Group		Sulfate, Nitrate				
Analytical Method		SW-846 9056A				
SOP Reference		TriMatrix SOP GR-02-113				
QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory 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.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Supervisor, QA Manager	Bias Contamination	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.
LCS containing sulfate and nitrate	One per preparatory batch	See Appendix C	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	Analyst, Supervisor, QA Manager	Accuracy Bias	See Appendix C
Matrix Spike	One per preparatory batch per matrix	See Appendix C	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	Analyst, Supervisor, QA Manager	Accuracy Bias	See Appendix C
Matrix Spike Duplicate	One per preparatory batch per matrix	See Appendix C	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	See Appendix C
Sample Duplicate	One per every 10 samples.	%D ≤ 10% (between sample and sample duplicate).	Correct problem and reanalyze sample and duplicate.	Analyst, Supervisor, QA Manager	Precision	%D ≤ 10% (between sample and sample duplicate).

Notes:

%D = percent difference
 DQO = daily quality objective
 LCS = Laboratory control sample
 LOQ = limit of quantitation

QA = quality assurance
 QC = quality assurance



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix		Groundwater				
Analytical Group		Dissolved Gasses (methane, ethane, ethane)				
Analytical Method		RSK 175				
SOP Reference		TriMatrix SOP GR-03-130				
QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analytes detected > 1/2 LOQ and > 1/10 the amount measured in any sample. Blank result must not otherwise affect sample results.	Re-prepare and analyze all associated samples. Discuss with client/qualify if re-analysis not feasible.	Analyst/Laboratory, Supervisor	Bias Contamination	No analytes detected > 1/2 LOQ and > 1/10 the amount measured in any sample. Blank result must not otherwise affect sample results.
LCS	One per preparatory batch of up to 20 samples	See Appendix C	Re-prepare and analyze all associated samples. Discuss with client/qualify if re-analysis not feasible.	Analyst/Laboratory, Supervisor	Accuracy Bias	See Appendix C
Matrix Spike	One per preparatory batch of up to 20 samples	See Appendix C	Examine results of LCS. If both the LCS and MS/MSD are unacceptable, re-prepare and analyze the associated samples and QC, otherwise report and narrate.	Analyst/Laboratory, Supervisor	Accuracy Bias	See Appendix C
Matrix Spike Duplicate	One per preparatory batch of up to 20 samples	See Appendix C	Examine results of LCS. If both the LCS and MS/MSD are unacceptable, re-prepare and analyze the associated samples and QC, otherwise report and narrate.	Analyst/Laboratory, Supervisor	Accuracy Bias Precision	See Appendix C

Notes:

- MS/MSD = matrix spike/matrix spike duplicate
- LCS = laboratory control sample
- LOQ = limit of quantitation
- QC = quality assurance
- SOP = Standard Operating Procedure



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SAP WORKSHEET #29: PROJECT DOCUMENTS AND RECORDS TABLE

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
<p>Field Documents Field Logbook Field Sample Forms Chain of Custody Records Air Bills Sampling Instrument Calibration Logs Sampling Notes Photographs SAP Health and Safety Plan</p>	<p>Field documents will be maintained in the project file located in the Resolution Consultants' Minneapolis, Minnesota office for 7 years after completion of contract task order.</p>
<p>Laboratory Documents Sample receipt, custody, and tracking record Equipment calibration logs Sample preparation logs Analysis Run logs Corrective Action forms Reported field sample results Reported results for standards, QC checks, and QC samples Extraction/clean-up records Raw data</p>	<p>Per Section 4.12, <i>Control of Records</i>, of the Department of Defense (DoD) Quality Systems Manual (2010), all analytical documents and records shall be retained by the laboratory for a minimum of 5 years. All information necessary for the historical reconstruction of data must be maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.</p> <p>Laboratory data deliverables will also be maintained in the Resolution Consultants' Minnesota project file.</p>
<p>Laboratory Electronic Deliverables Analytical Data</p>	<p>Analytical data will be compiled, validated and verified using the project database, an EQUIS database maintained by Resolution Consultants. The data is located on a password protected Structured Query Language server.</p> <p>The project database provides the data validator with the data to be validated and the electronic "form" for making validation corrections (qualifiers, results adjustments) which is uploaded back into the database to apply the validation corrections. Report tables, maps and other figures are prepared from the project database, to include the validation qualifiers.</p> <p>Once the verification is complete, it is exported into the NIRIS electronic data deliverable format for upload into the NIRIS database, maintained by the Navy.</p>



Document	Where Maintained
Assessment Findings Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (includes tabulated data summary forms)	All assessment documents will be maintained in the Resolution Consultants' Minneapolis, Minnesota project file and electronically in the server library.
Reports Source Area Investigation Report for Remedial System Optimization	All reports will be stored in hardcopy in the Resolution Consultants' Minneapolis, Minnesota project file, electronically in the server library, and will be uploaded to the NIRIS database. Hard copies and CD-ROMs of reports are also forwarded to NAVFAC and stored at the National Archives

Notes:

- NAVFAC = Naval Facilities Engineering Command
- NIRIS = Naval Installation Restoration Information Solution
- QC = Quality control
- SAP = Sampling and Analysis Plan

Long-Term Storage/Location of Project Documents (Listed Above)

Analytical data generated in the field and reports generated for the project will be stored in computer readable data files by AECOM Technical Services, Inc. in the Minneapolis office. As outlined in the Environmental Restoration Program Recordkeeping Manual, reports, analytical data, etc will be maintained in the Administrative Record File. Other files will be maintained at the office until completion of the tasks described in this SAP. Upon completion of the project, hardcopy files will be transferred to long-term data package storage at a third-party professional document storage firm and stored for 7 years.



SAP WORKSHEET #30: ANALYTICAL SERVICES TABLE

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method/SOP	Data Package TAT	Laboratory/Organization ¹ (name and address, contact person and telephone number)	Backup Laboratory
Groundwater	VOCs	See Worksheet #18	SW-846 5030/8260BGR-04-104	21 Calendar Days	TriMatrix Laboratories Inc., 5560 Corporate Exchange Court, SE, Grand Rapids, MI 49512 Walt Roudebush, 616-975-4561	None
Groundwater	Iron	See Worksheet #18	SW-846 3010A/6010C GR-01-147/GR-01-100	21 Calendar Days		
Groundwater	Sulfate/Nitrate	See Worksheet #18	SW-846 9056A GR-02-113	21 Calendar Days		
Groundwater	Dissolved Gases	See Worksheet #18	RSK 175 GR-03-130	21 Calendar Days		
Soil	VOCs	See Worksheet #18	SW-846 5035/8260B GR-04-104	21 Calendar Days		
Soil	Iron	See Worksheet #18	SW-846 3050B/6010C GR-01-100	21 Calendar Days		
Soil	TOC	See Worksheet #18	Walkley-Black GR-06-105	21 Calendar Days		
IDW Groundwater	TCLP VOCs	See Worksheet #18	SW8260B GR-03-105/GR-04-104	21 Calendar Days		
	TCLP SVOCs	See Worksheet #18	SW8270C GR-04-103	21 Calendar Days		
	TCLP Pesticides	See Worksheet #18	SW8081B GR-03-120	21 Calendar Days		
	TCLP Metals	See Worksheet #18	SW6010C/7470A GR-01-100/ GR-01-123	21 Calendar Days		
	PCBs	See Worksheet #18	SW8082A GR-03-128	21 Calendar Days		
	Ignitability	See Worksheet #18	SW1020A GR-18-124	21 Calendar Days		
	pH	See Worksheet #18	9040C GR-07-100	21 Calendar Days		



Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method/SOP	Data Package TAT	Laboratory/Organization ¹ (name and address, contact person and telephone number)	Backup Laboratory
IDW Soil	TCLP VOCs	See Worksheet #18	SW-846 1311/8260B GR-01-119/GR-04-104	21 Calendar Days		
	TCLP SVOCs	See Worksheet #18	SW-846 1311/8270C GR-01-119/GR-04-1	21 Calendar Days		
	TCLP Pesticides	See Worksheet #18	SW-846 1311/8081B GR-01-119/GR-03-120	21 Calendar Days		
	TCLP Metals	See Worksheet #18	SW-846 1311/6010C GR-01-119/GR-01-100/GR-01-128	21 Calendar Days		
	PCBs	See Worksheet #18	SW8082A GR-03-128	21 Calendar Days		
	Ignitability	See Worksheet #18	SW1020A GR-18-124	21 Calendar Days		
	pH	See Worksheet #18	SW9045D GR-07-113	21 Calendar Days		
	Paint Filter Test	See Worksheet #18	9095B GR-19-102	21 Calendar Days		

Notes:

¹ Laboratory meets accreditation requirements to support project needs.

- ID = identification
- IDW = investigation-derived waste
- PCB = polychlorinated biphenyl
- SOP = Standard Operating Procedure
- SVOC = semivolatle organic compounds
- TAT = turn-around time
- TCLP = toxicity characteristic leaching procedure
- TOC = total organic carbon
- VOC = Volatile organic compounds



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	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing CA	Person(s) Responsible for Monitoring Effectiveness of CA
Review of field procedures	Daily	Internal	Resolution Consultants	FOL, Resolution Consultants	PM, Resolution Consultants	PM, Resolution Consultants	PM, Resolution Consultants FOL, Resolution Consultants
Review of field notes	Daily	Internal	Resolution Consultants	FOL, Resolution Consultants	PM, Resolution Consultants	PM, Resolution Consultants	PM, Resolution Consultants Field Manager, Resolution Consultants
Review of field instrument calibration sheets	Daily	Internal	Resolution Consultants	FOL, Resolution Consultants	PC, Resolution Consultants	PC, Resolution Consultants	PM, Resolution Consultants FOL, Resolution Consultants
Review of COC forms	For each sample shipment	Internal/ External	Resolution Consultants / TriMatrix	PC, Resolution Consultants / Laboratory PM	PC, Resolution Consultants	Field Manager, Resolution Consultants PC, Resolution Consultants	PC, Resolution Consultants
Review of QC results and data package	For each data package	External/ Internal	TriMatrix / Resolution Consultants	Laboratory PC /PC	Laboratory PC, TriMatrix	Laboratory PC, TriMatrix	PC, Resolution Consultants

Notes:

- CA = corrective action
- COC = chain-of-custody
- FOL = field operations leader
- PC = Project Chemist
- PM = Project Manager
- QC = quality control



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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) ^a	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization) ^a	Timeframe for Response
Review of field procedures	Non-conformance Form (NC)	PM, Resolution Consultants	24 hours after audit (no notification required if no deficiencies observed)	Verbal Communication/logbook record	FOL, Resolution Consultants	24 hours after notification
Review of field notes	Non-conformance Form (NC)	PM, Resolution Consultants	24 hours after audit (no notification required if no deficiencies observed)	Verbal Communication/logbook record	FOL, Resolution Consultants	24 hours after notification
Review of field instrument calibration sheets	Non-conformance Form (NC)	PC, Resolution Consultants PM, Resolution Consultants	24 hours after audit (no notification required if no deficiencies observed)	Verbal Communication/logbook record	FOL, Resolution Consultants	24 hours after notification
Review of COC forms	email notice of non-conformance, Laboratory case narrative	PC Resolution Consultants PM, Resolution Consultants	24 hours after audit (no notification required if no deficiencies observed)	Verbal Communication/email documentation	FOL, Resolution Consultants PM, Resolution Consultants	24 hours after notification
Review of QC results and data package	email notice of non-conformance, Laboratory case narrative	PC, Resolution Consultants PM, Resolution Consultants	Non-conformance notification – within 24 hour of observation. Case narrative, within 21 days of sample collection	Verbal Communication/email documentation	PM, Resolution Consultants	24 hours after notification

Notes:

- ^a = Individuals identified in Worksheet #7.
- COC = chain-of-custody
- FOL = Field Operations Leader
- PC = Project Chemist
- PM = Project Manager
- QC = quality control



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)
Chemical Data Quality Review Report	Per sample delivery group	Within 4 weeks after receiving the data from the laboratory	PC or Data Validator, Resolution Consultants	PM, Resolution Consultants; project file
Laboratory Quality Assurance Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, TriMatrix	PC, PM, and project file, Resolution Consultants
Source Area Investigation Report for Remedial System Optimization	Upon project completion	See Worksheet #16	PM, Resolution Consultants	RPM, Department of the Navy NAVFAC MW RPM, USEPA Region 5 PM, MPCA

Notes:

- MPCA = Minnesota Pollution Control Agency
- NAVFAC MW = Naval Facilities Engineering Command, Midwest
- PC = Project Chemist
- PM = Project Manager
- RPM = Remedial Project Manager
- USEPA = United States Environmental Protection Agency



SAP WORKSHEETS #34-36: DATA VERIFICATION AND VALIDATION (STEPS I AND IIA/IIB) PROCESS TABLE
 (UFP-QAPP Manual Section 5.2.1)

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/IIb	Internal/External
Verification Chain-of-custody forms Sample Login/Receipt	Review the sample shipment for completeness, integrity, and sign accepting the shipment. All sample labels will be checked against the chain-of-custody form, and any discrepancies will be identified, investigated, and corrected. The samples will be logged in at every storage area and work station required by the designated analyses. Individual analysts will verify the completeness and accuracy of the data recorded on the forms.	Laboratory sample custodians and analysts, TriMatrix	I	Internal
Verification Chain-of-custody forms	Check that the chain-of-custody form was signed/dated by the sampler relinquishing the samples and by the laboratory sample custodian receiving the samples for analyses.	Project chemist or data validators, Resolution Consultants	I	External
Verification SAP sample tables	Verify that all proposed samples listed in the SAP tables have been collected.	FOL or designee, Resolution Consultants	I	External
Verification Sample log sheets and field notes	Verify that information recorded in the log sheets and field notes are accurate and complete.	FOL or designee, Resolution Consultants	I	External
Verification Field QC samples	Check that field QC samples, described in Worksheet #12 and listed in Worksheet #20 were collected as required.	FOL or designee, Resolution Consultants	I	External
Verification Analytical data package	Verify all analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory project manager (or designee) will sign the case narrative for each data package.	Laboratory project manager, TriMatrix	I	Internal
Verification Analytical data package	Verify the data package for completeness. Missing information will be requested from the laboratory and validation (if performed) will be suspended until missing data are received.	FOL, Project chemist or data validators, Resolution Consultants	I	External
Verification Electronic data deliverables	Verify the electronic data against the chain-of-custody and hard copy data package for accuracy and completeness.	Data manager and/or validator, Resolution Consultants	I	External



Data Review Input	Description	Responsible for Verification (name, organization)	Step I/ IIa/IIb ¹	Internal/ External
Validation Chain-of-custody	Examine the traceability of the data from time of sample collection until reporting of data. Ensure that the custody and integrity of the samples were maintained from collection to analysis and the custody records are complete and any deviations are recorded.	Project chemist or data validators, Resolution Consultants	IIa	External
Validation Holding Times	Review that the samples were shipped and stored at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times. If holding times were not met, confirm that deviations were documented.	Project chemist or data validators, Resolution Consultants	IIa	External
Validation Sample results for representativeness	Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Laboratory data results for accuracy	Ensure that the laboratory QC samples were analyzed and that the measurement performance criteria, listed in Worksheet #28, were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed, as listed in Worksheet #12, and that the analytical QC criteria were met.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Field and laboratory duplicate analyses for precision	Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSDs. Ensure compliance with the precision goals listed in Worksheets #12 and #28.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Project action limits	Assess and document the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant, on the other target compounds reported as undetected.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Data quality assessment report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all the data qualifications. Present tabular qualified data and data qualifier codes and summarize data qualification outliers. Determine if the data met the measurement performance criteria and determine the impact of any deviations on the technical usability of the data.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External



Data Review Input	Description	Responsible for Verification (name, organization)	Step I/ IIa/IIb ¹	Internal/ External
Validation SAP QC sample documentation	Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within acceptance limits.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Analytical data deviations	Determine the impact of any deviation from sampling or analytical methods and laboratory SOP requirements and matrix interferences effect on the analytical results.	Project chemist or data validators, Resolution Consultants	IIb	External
Validation Project quantitation limits for sensitivity	Ensure that the project detection limits were achieved.	Project chemist or data validators, Resolution Consultants	IIb	External
Validation Groundwater and Soil — VOCs	Validate VOC data using SW846 8260B method-specific criteria, data quality indicators provided in the DoD QSM, and those listed in Worksheets #12, #19, and #28. All data will be validated and raw instrument outputs assessed and recalculated for 10% of the reported results. USEPA's <i>Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review</i> (2008) will be used for the general approach in applying qualifiers. However, qualifiers will be assigned when control limits identified in this SAP are exceeded rather than using the control limits in the National Functional Guidelines.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External



Data Review Input	Description	Responsible for Verification (name, organization)	Step I/ IIa/IIb ¹	Internal/ External														
Validation Data qualifiers	Qualifiers that will be applied during the data validation process are summarized below and, as indicated, results will be considered usable for interpretation unless the results are rejected when extreme data quality indicator failures are noted. The Data Validation Checklists in Appendix B summarize procedures for assigning data qualifiers.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External														
	<table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="text-align: left;">Qualifier</th> <th style="text-align: left;">Definition</th> </tr> </thead> <tbody> <tr> <td>J</td> <td>Estimated value because result is at a concentration less than the LOQ or due to associated QC outliers</td> </tr> <tr> <td>UJ</td> <td>Estimated reporting limit due to associated QC outliers</td> </tr> <tr> <td>B</td> <td>Analyte result is considered a high estimated value due to contamination present in an associated blank.</td> </tr> <tr> <td>UB</td> <td>Detected result is less than the limit of quantitation and the detection is considered to be a false positive due to contamination present in an associated blank.</td> </tr> <tr> <td>R</td> <td>Analyte result is rejected – result is not usable.</td> </tr> <tr> <td>NP</td> <td>A second, more technically valid result is available (to be used if more than one result is reported for an analyte per method).</td> </tr> </tbody> </table>				Qualifier	Definition	J	Estimated value because result is at a concentration less than the LOQ or due to associated QC outliers	UJ	Estimated reporting limit due to associated QC outliers	B	Analyte result is considered a high estimated value due to contamination present in an associated blank.	UB	Detected result is less than the limit of quantitation and the detection is considered to be a false positive due to contamination present in an associated blank.	R	Analyte result is rejected – result is not usable.	NP	A second, more technically valid result is available (to be used if more than one result is reported for an analyte per method).
	Qualifier				Definition													
	J				Estimated value because result is at a concentration less than the LOQ or due to associated QC outliers													
	UJ				Estimated reporting limit due to associated QC outliers													
	B				Analyte result is considered a high estimated value due to contamination present in an associated blank.													
	UB				Detected result is less than the limit of quantitation and the detection is considered to be a false positive due to contamination present in an associated blank.													
R	Analyte result is rejected – result is not usable.																	
NP	A second, more technically valid result is available (to be used if more than one result is reported for an analyte per method).																	

Notes:

- ¹IIa = Compliance with methods, procedures, and contracts [see Table 10, page 117, UFP-QAPP Manual, V.1, March 2005.]
- ¹IIb = Comparison with measurement performance criteria in the SAP [see Table 11, page 118, UFP-QAPP Manual, V.1, March 2005]
- SAP = Sampling and analysis plan
- FOL = Field team leader
- LOQ = limit of quantitation
- QC = Quality control
- RPD = Relative percent difference
- MS/MSD = Matrix spike/Matrix Spike duplicate
- LCS/LCSD = Laboratory control sample/laboratory control sample duplicate
- SOP = Standard operating procedure
- VOCs = Volatile organic compounds
- DoD QSM = *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2, October 2010
- UFP-QAPP = *Uniform Federal Policy for Quality Assurance Plans*
- USEPA = U.S. Environmental Protection Agency

SAP WORKSHEET #37: USABILITY ASSESSMENT

(UFP-QAPP Manual Section 5.2.3)

Data Review

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 Chemical Data Quality Review Report which will be appended to the final project report. Overall trends if any that may be associated with the data will be evaluated and discussed in the Chemical Data Quality Review Report.

- **Completeness** — The PC, acting on behalf of the Project Team, will determine whether deviations from the scheduled sample collection or analyses occurred. If they have occurred and the Resolution Consultants PM determines that the deviations compromise the ability to meet project objectives she will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Completeness will be measured by determining the percentage of usable results out of planned results. Usable results are results that have not been rejected during data review and validation. Planned results are all the results that were planned to be reported for the project or a method.

$$\text{Completeness} = \frac{\text{Usable Results} \times 100}{\text{Planned Results}}$$

The completeness goal for samples received at the laboratory for this project is 95 percent.

The completeness goal for the collection of planned field samples is 80 percent. It is considered possible that site conditions will prevent the collection of all samples, particularly samples at greater depths.

- **Precision** — The PC, 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 laboratory duplicate results will be no less precise than field duplicate results. If the goals are not met or data have been flagged as estimated (Q qualifier) limitations on the use of the data will be described in the project report.
- **Accuracy** — The PC, 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, MS/MSD, and LCS. 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 Resolution Consultants 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 analyzed in accordance with this 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. Verify that standardized SOPs were followed for the collection of field samples and field measurements.
- **Comparability** — The PC, acting on behalf of the Project Team, will determine whether the data generated under this project are sufficiently comparable to historical property data generated by different methods and for samples collected using different procedures and under different property conditions. This will not require quantitative comparisons unless the Project Chemist indicates that such quantitative analysis is required. Verify that standardized SOPs were followed for the installation of monitoring wells, collection of field samples, and field measurements. MIP results for VOCs will be compared to definitive groundwater and soil results to determine how well the screening procedure can correlate with the definitive results.
- **Sensitivity** — The PC, acting on behalf of the Project Team, will determine whether project sensitivity goals listed in Worksheet #15 are achieved. If sensitivity goals are not achieved, the limitations on the data will be described.

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. The Resolution Consultants FOL, PC, and 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. Rejected data will not be used for making decisions.



Identify the personnel responsible for performing the usability assessment:

The Resolution Consultants PM, PC, and FOL will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Project Team. 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, B, UB) or rejection (R). The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary. Graphical presentations of the data such as concentration tag maps will be generated as part of the overall data evaluation process.



REFERENCES

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RMT Inc. (1991, February). A-E Quality Control Summary Report For Well Installation and Ground Water and Soil Sampling at the Naval Industrial Reserve Ordnance Plant Fridley, Minnesota.

Tetra Tech NUS Inc. (2002, April). Remedial Investigation for Operable Unit 3 , Naval Industrial Reserve Ordnance Plant Fridley, Minnesota.

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USEPA (2005, March). Uniform Federal Policy for Quality Assurance Plans Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs, Parts 1, 2A, 2B, and 2C. (Final Version 1), USEPA-505-B-04-900A.

USEPA (2006, February). *Guidance on Systematic Planning Using the Data Quality Objectives Process.* USEPA QA/G-4. USEPA/240/B-06/001. Office of Environmental Information.

USEPA (2008, June). USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review. Office of Solid Waste and Emergency Response, USEPA-540-R-08-01.



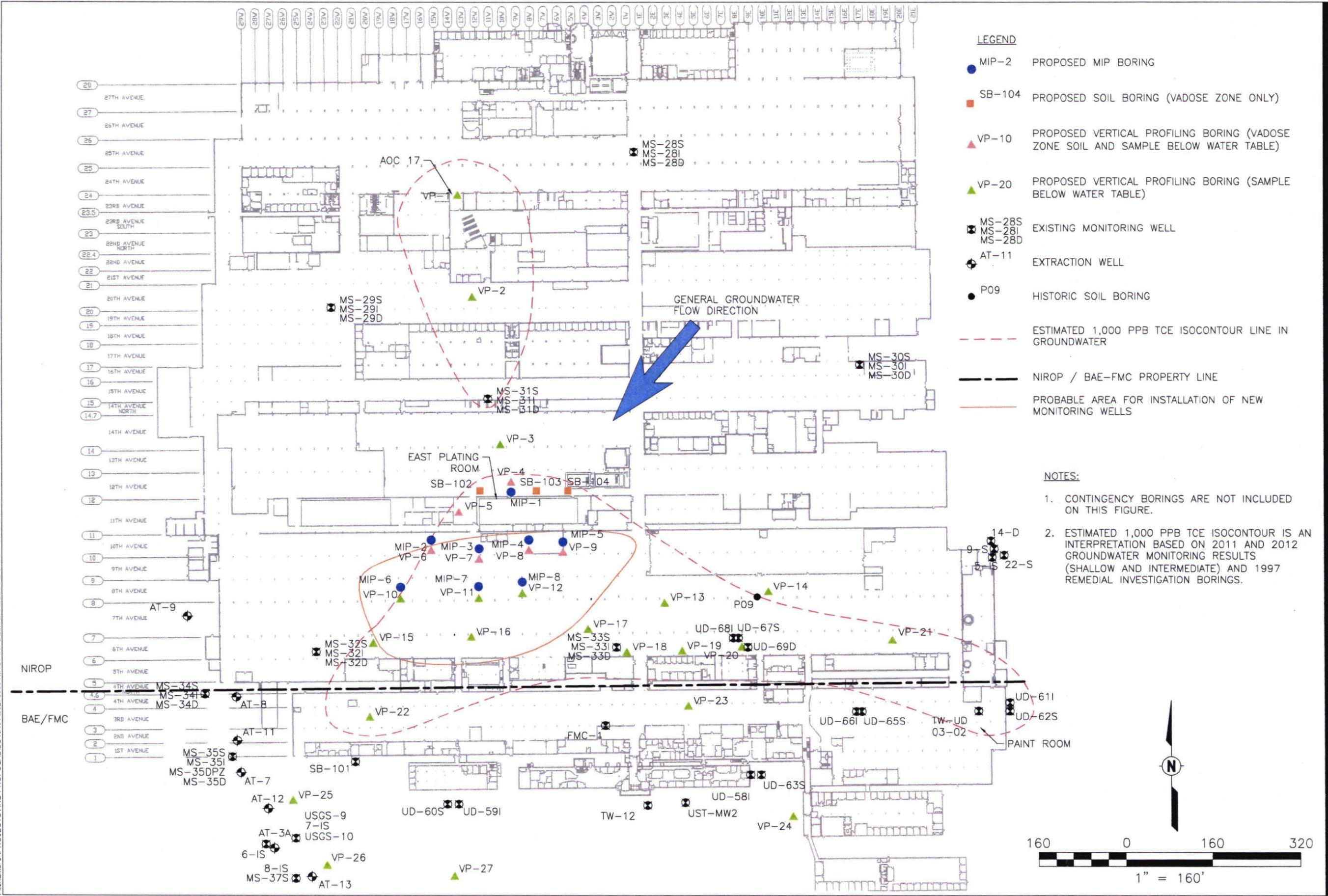
**RESOLUTION
CONSULTANTS**

*Sampling and Analysis Plan — Source Area Investigation
Naval Industrial Reserve Ordnance Plant, Fridley Minnesota*

References

Revision: 0; July 2013

USEPA (2010, January). USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review. Office of Solid Waste and Emergency Response, 9240.1-51; USEPA 540-R-10-011.

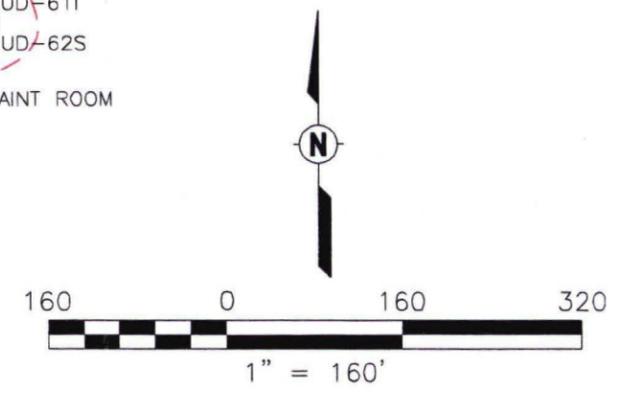


LEGEND

- MIP-2 PROPOSED MIP BORING
- SB-104 PROPOSED SOIL BORING (VADOSE ZONE ONLY)
- ▲ VP-10 PROPOSED VERTICAL PROFILING BORING (VADOSE ZONE SOIL AND SAMPLE BELOW WATER TABLE)
- ▲ VP-20 PROPOSED VERTICAL PROFILING BORING (SAMPLE BELOW WATER TABLE)
- ⊗ MS-28S, MS-28I, MS-28D EXISTING MONITORING WELL
- ⊕ AT-11 EXTRACTION WELL
- P09 HISTORIC SOIL BORING
- - - ESTIMATED 1,000 PPB TCE ISOCONTOUR LINE IN GROUNDWATER
- - - NIROP / BAE-FMC PROPERTY LINE
- PROBABLE AREA FOR INSTALLATION OF NEW MONITORING WELLS

NOTES:

1. CONTINGENCY BORINGS ARE NOT INCLUDED ON THIS FIGURE.
2. ESTIMATED 1,000 PPB TCE ISOCONTOUR IS AN INTERPRETATION BASED ON 2011 AND 2012 GROUNDWATER MONITORING RESULTS (SHALLOW AND INTERMEDIATE) AND 1997 REMEDIAL INVESTIGATION BORINGS.



APPENDIX A
REFERENCE FIGURES FROM PREVIOUS REPORTS

Figure A-1. Source:
 Record of Decision Operable Unit (OU) 2 and Operable Unit (OU) 3, NIROP, Fridley, MN, Southern
 Division Naval Facilities Engineering Command, Contract No. N62467-94-D-0888, CTO 0003,
 August 2003

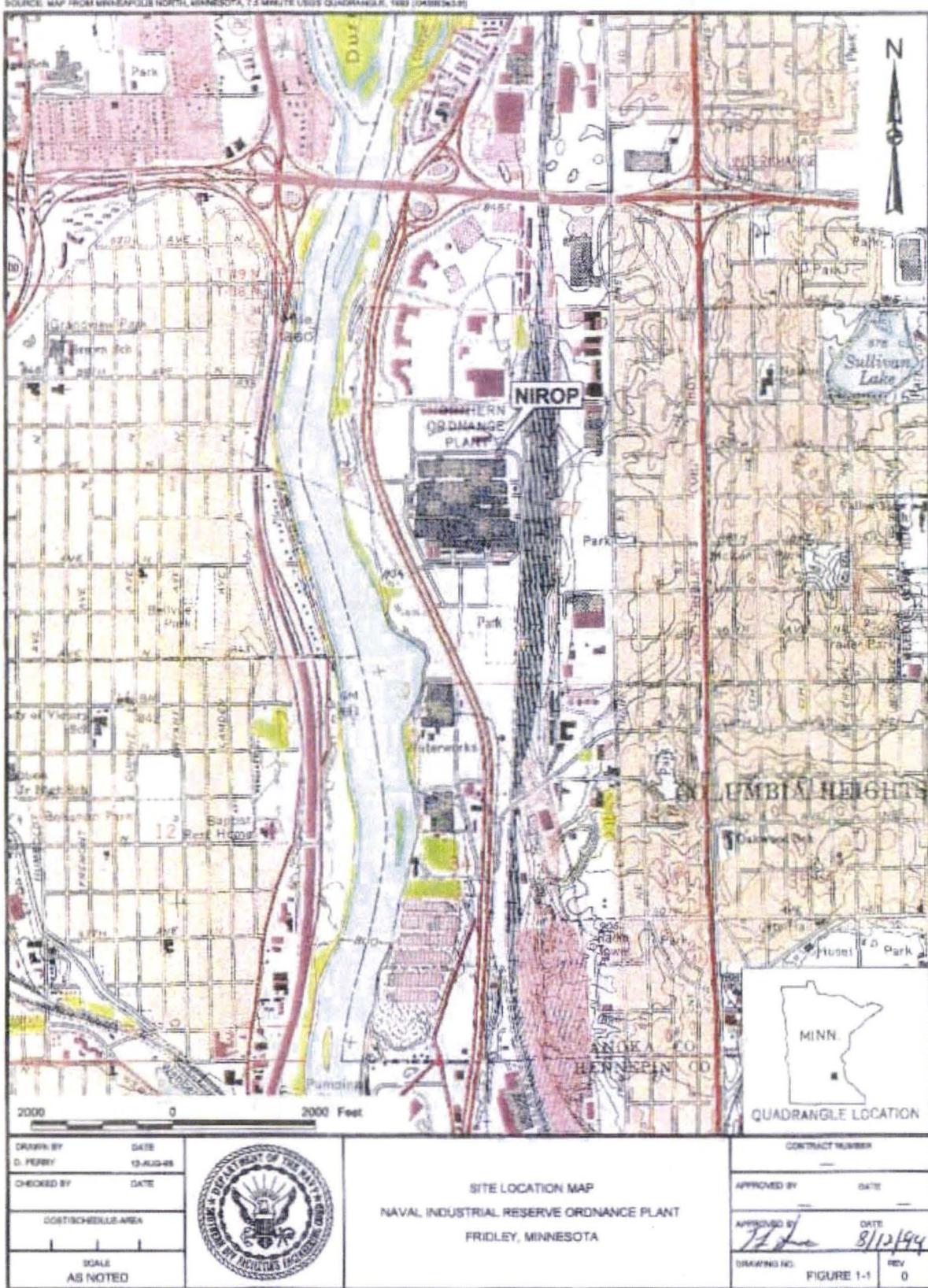


FIGURE 1-1: NIROP SITE ASSESSMENT APR. 13-AUG-88 DNP LOCATION MAP LAYOUT

Figure A-2. Source:
2011 Annual Monitoring Report, NIROP, Fridley, MN, Tetra Tech, November 2012

PGH P:\GIS\FRIDLEY_NIROP\MAPDOCS\MXD\2011_AMR_MONITORING_WELLS.MXD 06/13/12 JEE

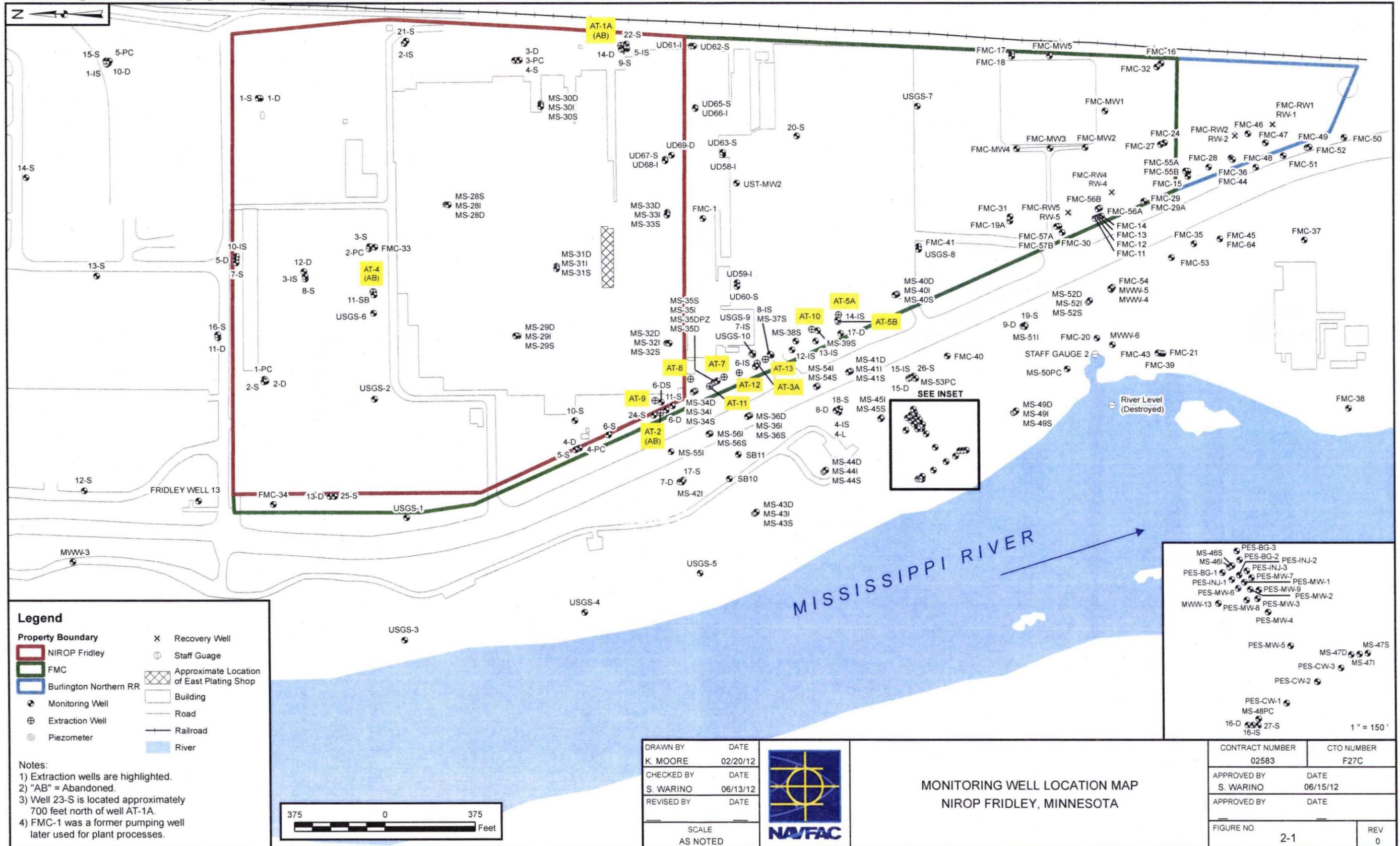
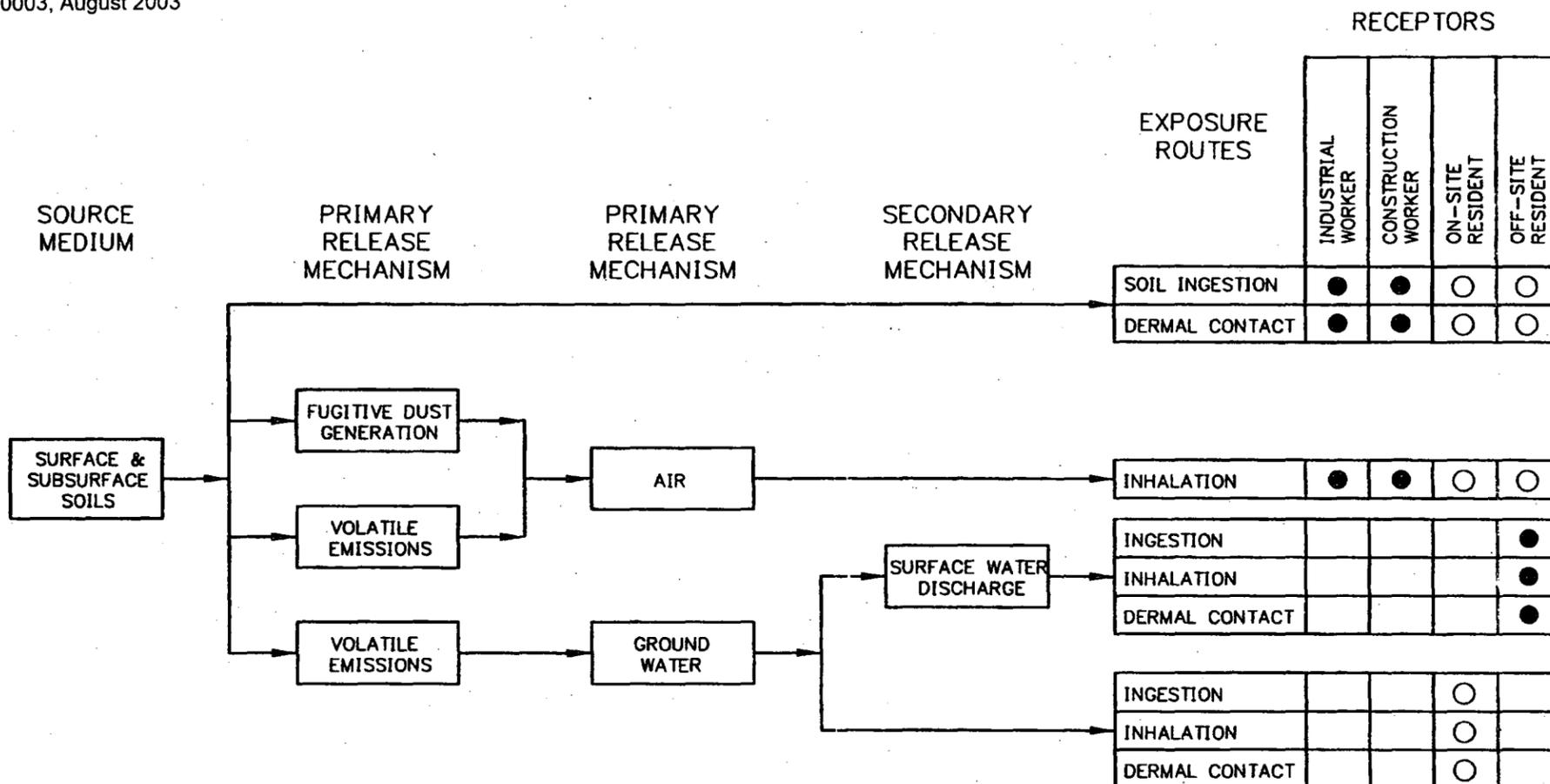


Figure A-3. Source:
 Record of Decision Operable Unit (OU) 2 and Operable Unit (OU) 3,
 Southern Division Naval Facilities Engineering Command, Contract
 No. N62467-94-D-0888, CTO 0003, August 2003

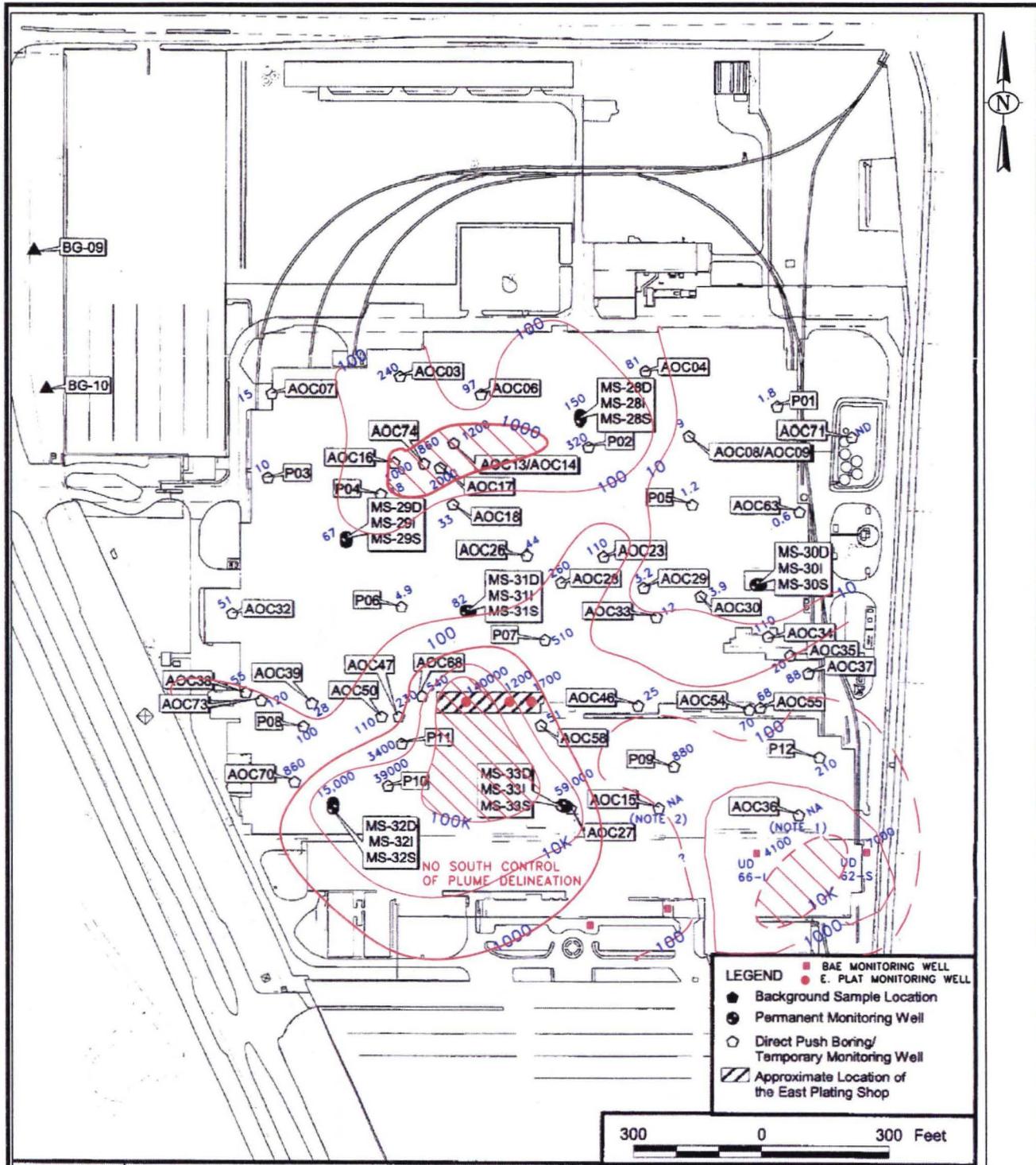


LEGEND:

- POTENTIALLY COMPLETE EXPOSURE PATHWAY
- INCOMPLETE HYPOTHETICAL EXPOSURE PATHWAY

NO.	DATE	REVISIONS	BY	CHKD	APPD	REFERENCES	DRAWN BY	DATE		CONTRACT NO. 6966	
							MF	5/10/00			
							CHECKED BY	DATE			
							COST/SCHED-AREA				
							SCALE	NONE	CONCEPTUAL SITE MODEL OPERABLE UNIT 3 NIROP, FRIDLEY	APPROVED BY	DATE
										APPROVED BY	DATE
										DRAWING NO.	REV.
										FIGURE 2-6	0

Figure A-4. Source: Source Investigation Map Methodology, NIROP Superfund Site, Fridley, MN, NAFVAC MW, February 2012



LEGEND

2000 TCE CONCENTRATION PPB
 NOTE 1: WELL SCREENED IN CLAY
 NOTE 2: PUSHED POINT, MECH. PROBLEMS
 NOTE 3: GW SAMPLE FROM LONGER SCREEN, DEEPER INTERVAL

= PROBABLE SOURCE AREA

BASEMAP AND DATA FROM NIROP RI, TETRA-TECH, 2002(1997 DATA) & RCRA RPT, BAE SYSTEMS, 2007

TITLE: FIGURE 2A: SOURCE AREA INVESTIGATION
 TCE CONCENTRATION GROUNDWATER-DEPTH INDEPENDENT
 NIROP FRIDLEY, MINNESOTA

CAD: HP DATE: 12/29/11 FOR:
 APPROVED: NAVFAC SCALE: AS SHOWN



FOR: NAVFAC MIDWEST
 201 DECATUR AVENUE
 GREAT LAKES, ILLINOIS

PROJECT NO. NIROP
 DRAWING NO. GW SOURCE 2

APPENDIX B
RESOLUTION CONSULTANTS STANDARD OPERATING PROCEDURES,
FIELD FORMS, AND DATA VALIDATION CHECKLISTS



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 purposes 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.

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 of 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 with 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.

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;

- 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)	<p>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.</p> <p>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.</p>
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.

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.

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- 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

Attachment 1 Chain-of-Custody Seal

CHAIN-OF-CUSTODY SEAL

<i>[LABORATORY]</i>	SAMPLE NO.	DATE	SEAL BROKEN BY
	SIGNATURE		DATE
	PRINT NAME AND TITLE (<i>Inspector, Analyst or Technician</i>)		

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

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 7.2.2 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 (HNO₃), sulfuric acid (H₂SO₄), 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 7.2.1.

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 7.2.1.



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

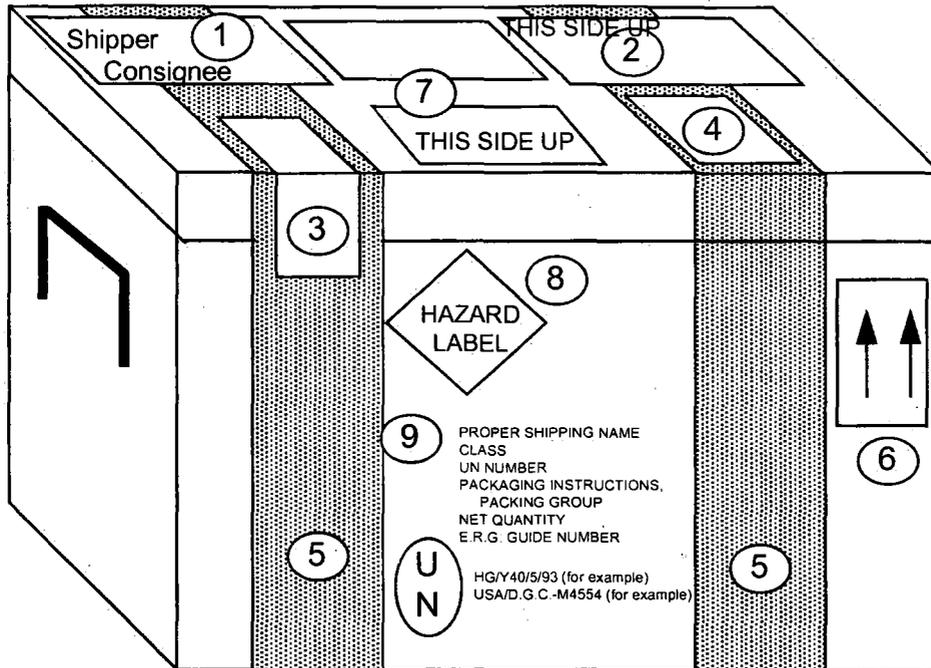
8.9 Attachment 9 – Soil Samples Restricted Entry Labels

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

Attachment 1 Example Hazardous Material Package Marking



- | | |
|--|---|
| ① AIR BILL/COMMERCIAL INVOICE | ⑥ DIRECTION ARROWS STICKER - TWO REQUIRED |
| ② USDA PERMIT (Letter to Laboratory from USDA) | ⑦ THIS SIDE UP STICKERS |
| ③ CUSTODY SEAL | ⑧ HAZARD LABEL |
| ④ USDA 2" X 2" SOIL IMPORT PERMIT | ⑨ HAZARDOUS MATERIAL INFORMATION |
| ⑤ WATERPROOF STRAPPING TAPE | ⑩ PACKAGE SPECIFICATIONS |

Attachment 2 Packing Groups

PACKING GROUP OF THE SUBSTANCE CLASS or DIVISION of PRIMARY or SUBSIDIARY RISK	PACKING GROUP I		PACKING GROUP II		PACKING GROUP III	
	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.

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
	<input type="checkbox"/>						
and the applicable UN Numbers are:							

Attachment 4 SW-846 Preservative Exception

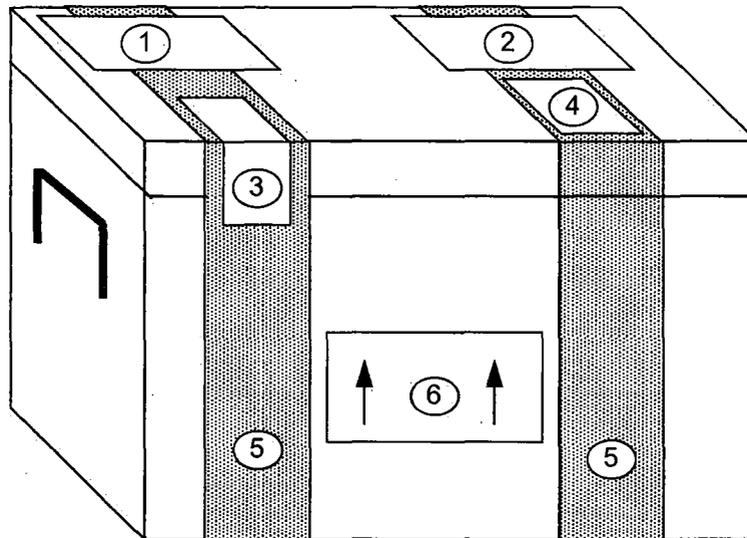
Measurement	Vol. Req. (mL)	Container ²	Preservative ^{3,4}	Holding Time ⁵
MBAS	250	P, G	Cool, 4°C	48 Hours
NTA	50	P, G	Cool, 4°C	24 Hours

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 4°C 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 (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) 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 <LaName> <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	cooler	Soil samples for laboratory analy				\$1.00	\$3.00
						TOTAL WEIGHT		TOTAL INVOICE VALUE
								\$3.00
								Check one <input type="checkbox"/> F.O.B. <input type="checkbox"/> C&F <input type="checkbox"/> 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

Attachment 8
Soil Import Permit



UNITED STATES
DEPARTMENT OF
AGRICULTURE

Animal and Plant
Health Inspection
Service

Plant Protection and
Quarantine

Soil Permit

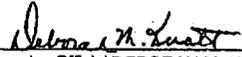
Permit
Number: S-52299

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. § 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. § 1001).

PPQ FORM 525B (6/94)

Pt. 1 - PERMITTEE

Attachment 9 Soil Samples Restricted Entry Labels

<hr/> <p>U.S. DEPARTMENT OF AGRICULTURE</p> <p>ANIMAL AND PLANT HEALTH INSPECTION</p> <p>SERVICE</p> <p>PLANT PROTECTION AND QUARANTINE</p> <p>HYATTSVILLE, MARYLAND 20782</p> <p>SOIL SAMPLES</p> <p>RESTRICTED ENTRY</p> <hr/>
<p>The material contained in this package is imported under authority of the Federal Plant Pest Act of May 23, 1957.</p> <hr/>
<p>For release without treatment if addressee is currently listed as approved by Plant Protection and Quarantine.</p> <hr/>
<p>PPQ FORM 550 <i>Edition of 12/77 may be used</i></p> <p>(JAN 83)</p>

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.

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.

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

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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.

- 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

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*.



Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue

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.

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.

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)

- 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 geotextile 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

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.

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 placed 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

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 variety 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.

- 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

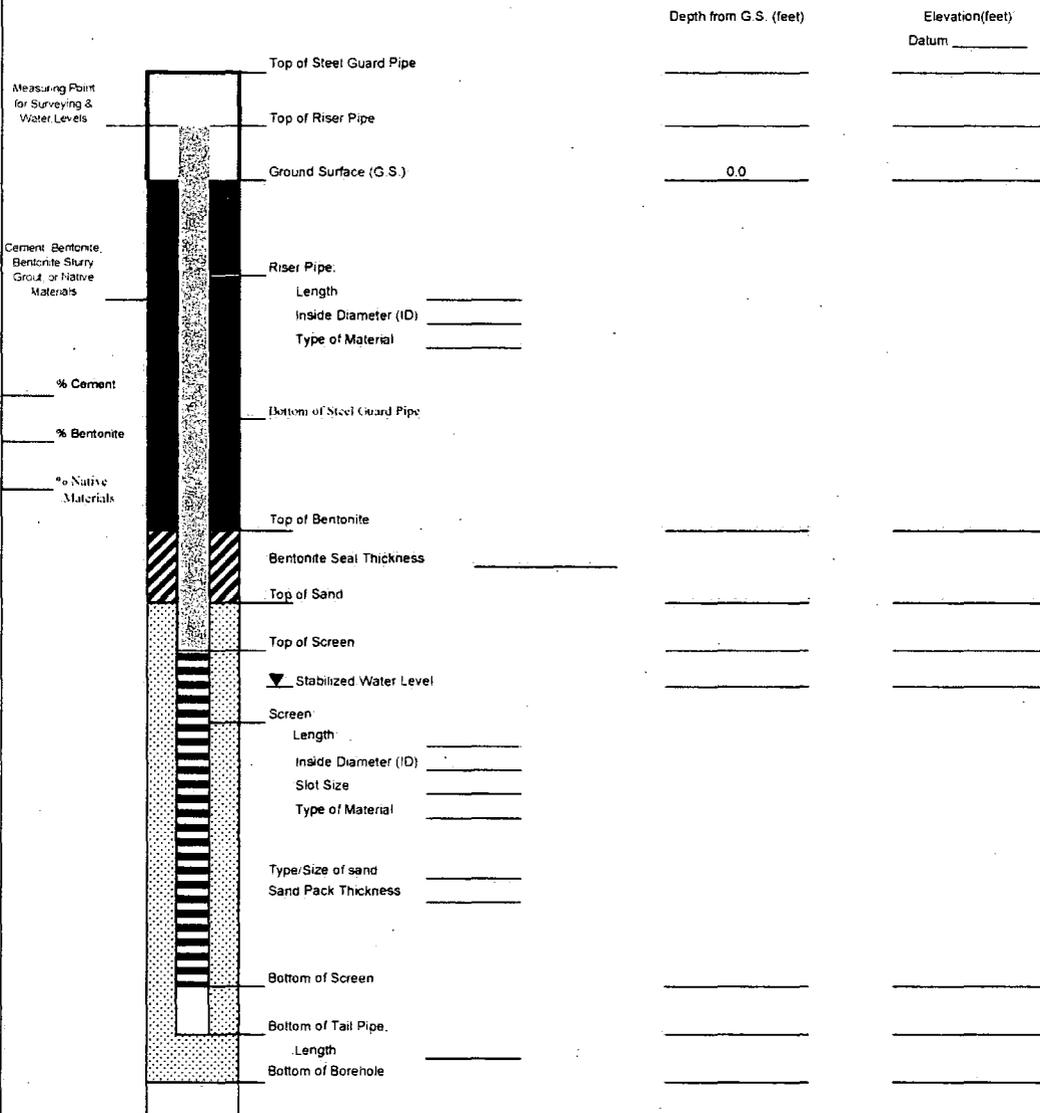
- 10.1 Attachment 1 – Monitoring Well Construction Form



- 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)

Attachment 1 Monitoring Well Construction Form

	Client: _____		WELL ID: _____	
	Project Number: _____		Date installed: _____	
	Site Location: _____		Inspector: _____	
	Well Location: _____	Coords: _____	Contractor: _____	
	Method: _____			
MONITORING WELL CONSTRUCTION DETAIL				
				
			Depth from G.S. (feet)	Elevation(feet)
			Datum _____	
Measuring Point for Surveying & Water Levels			_____	_____
Top of Steel Guard Pipe			_____	_____
Top of Riser Pipe			_____	_____
Ground Surface (G.S.)			0.0	_____
Riser Pipe:				
Length _____				
Inside Diameter (ID) _____				
Type of Material _____				
Bottom of Steel Guard Pipe			_____	_____
Top of Bentonite			_____	_____
Bentonite Seal Thickness _____				
Top of Sand			_____	_____
Top of Screen			_____	_____
▼ Stabilized Water Level			_____	_____
Screen				
Length _____				
Inside Diameter (ID) _____				
Slot Size _____				
Type of Material _____				
Type/Size of sand _____				
Sand Pack Thickness _____				
Bottom of Screen			_____	_____
Bottom of Tail Pipe:				
Length _____				
Bottom of Borehole			_____	_____
Borehole Diameter _____				
Approved: _____				
Describe Measuring Point: _____			Signature _____	Date _____

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.

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

- 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.

- 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.

- 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® system 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 discharge 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.

- Groundwater parameters for three consecutive standing water volumes are within the following:
 - pH – within ± 0.2 units
 - Specific conductivity – within $\pm 3\%$
 - ORP – within ± 10 mV
 - Temperature – within ± 1 degree Celsius
 - Turbidity – at or below 10 nephelometric turbidity units (NTU) or within $\pm 10\%$ 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.



7.6 Temporary Well Points

For certain projects, temporary well points (TWP) 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*.

<i>Author</i>	<i>Reviewer</i>	<i>Revisions (Technical or Editorial)</i>
Shawn Dolan Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (June 2012)



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	Has required volume been removed	Yes <input type="checkbox"/>	No <input type="checkbox"/>	N/A <input type="checkbox"/>
Maximum Turbidity Allowed _____ NTUs	Has required turbidity been reached	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Stabilization of parameters _____ %	Has parameters stabilized	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

If no or N/A explain below:

Signature _____ Date: _____

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 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.

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

- 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.

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.

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.



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 $\pm 10\%$ 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



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.

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.

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)

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 gases. 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

- 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*.

<i>Author</i>	<i>Reviewer</i>	<i>Revisions (Technical or Editorial)</i>
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)



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 (m³/min)	Drawdown (m)	Color/Odor/etc.

d. Acceptance criteria pass/fail Yes No N/A (continued on back)
 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 _____



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:

GW¹ Well graded (poorly sorted) gravel (>50 percent gravel, <5percent fines)

GP¹ Poorly graded (well sorted) gravel (>50percent gravel, <5percent fines)

GM¹ Silty gravel (>50 percent gravel, >15 percent silt)

GC¹ Clayey gravel (>50 percent gravel, >15 percent clay)

SW¹ Well graded (poorly sorted) sand (>50 percent sand, <5 percent fines)

SP¹ 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.

- SM¹ Silty sand (>50 percent sand, >15 percent silt)
- SC¹ Clayey sand (>50 percent sand, >15 percent clay)
- ML² Inorganic, low plasticity silt (slow to rapid dilatancy, low toughness, and plasticity)
- CL² Inorganic, low plasticity (lean) clay (no or slow dilatancy, medium toughness and plasticity)
- MH² Inorganic elastic silt (no to slow dilatancy, low to medium toughness and plasticity)
- CH² 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

Figure 8-1 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."

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.

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
		GRAVELS With Fines		GP	Poorly graded gravels, gravel-sand mixtures, little or no 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
		SANDS With Fines		SP	Poorly graded sands, gravelly sands, little or no 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)

GROUP SYMBOL

GROUP NAME

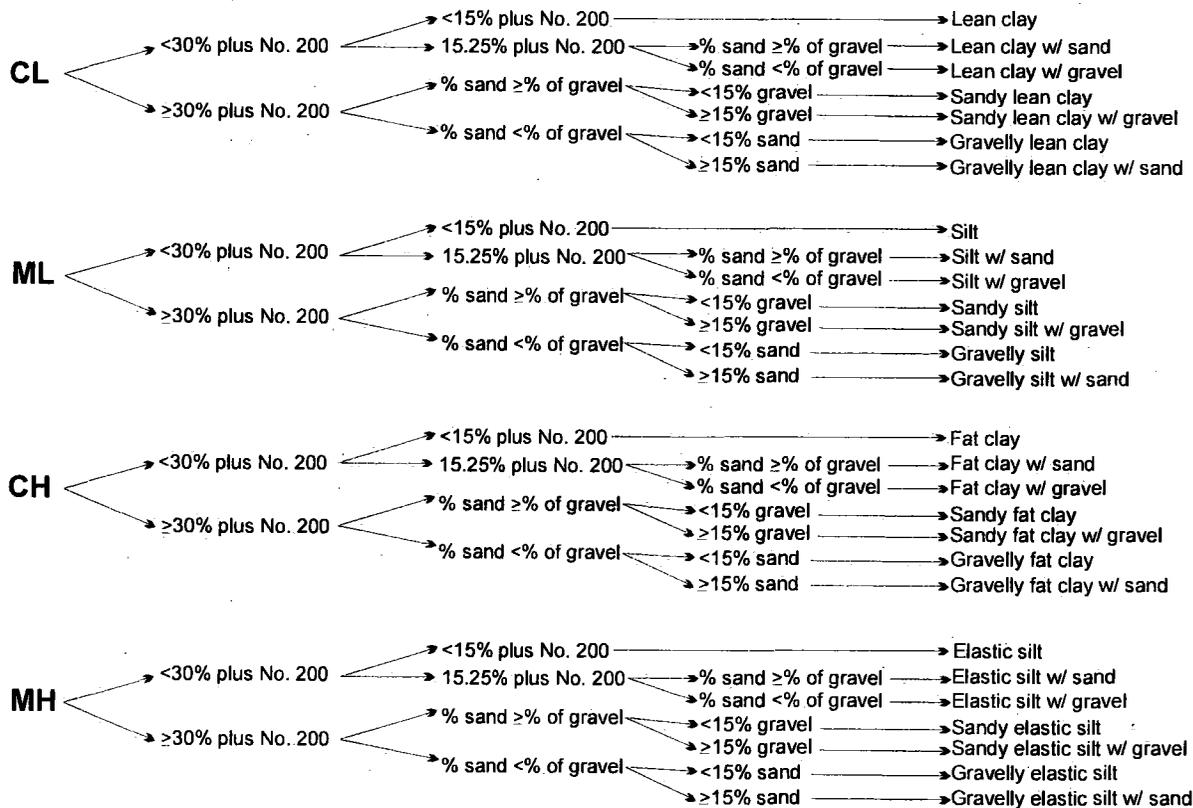
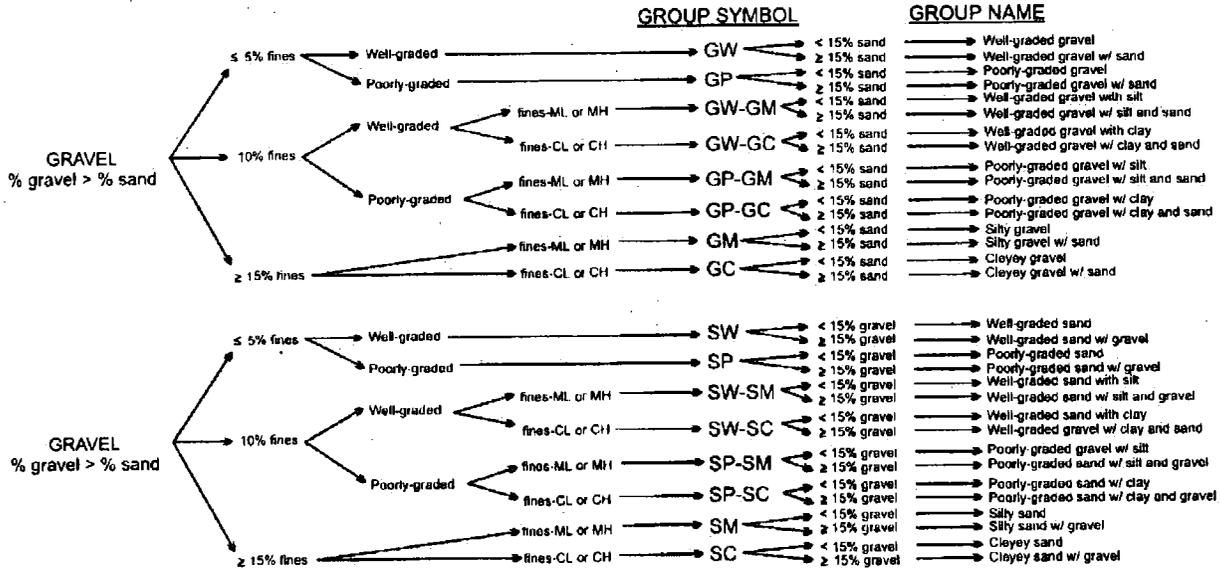


Figure 8-2: Flow Chart for Fine Grain Soil Classification



Figure 8-3: Flow Chart for Soil with Gravel



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.

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.

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

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

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, aplitic, 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



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

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Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue

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

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.



- 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)

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.

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.

- 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 $\pm 10\%$. 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;



- 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)

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.

- 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.

- 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

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 $\frac{3}{4}$ 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.

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)



<i>Client:</i>	BORING ID:	
<i>Project Number:</i>		
<i>Site Location:</i>		
<i>Coordinates:</i>		<i>Elevation:</i>
<i>Drilling Method:</i>		<i>Monitoring Well Installed:</i>
<i>Sample Type(s):</i>	<i>Boring Diameter:</i>	<i>Screened Interval:</i>

<i>Weather:</i>	<i>Logged By:</i>	<i>Date/Time Started:</i>	<i>Depth of Boring:</i>
<i>Drilling Contractor:</i>	<i>Ground Elevation:</i>	<i>Date/Time Finished:</i>	<i>Water Level:</i>

Depth (ft)	Casing Info	Annular Space Info	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:	<i>Date</i>	<i>Time</i>	<i>Depth to groundwater while drilling</i>

Checked by _____ Date: _____

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.

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.
pH	SU	0	14	5	9	DO less than 1 mg/L, ORP negative, may have sulfur odor, sulfide, ferrous iron and/or gray color. 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 ($\mu\text{S}/\text{cm}$) 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 $\mu\text{S}/\text{cm}$, 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 Factors

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.



$$K = \frac{(Km)(C)}{1 + 0.0191(T - 25)}$$

Where:

- K = Conductivity in $\mu\text{mhos/cm}$ at 25°C
- Km = Measured conductivity in $\mu\text{mhos/cm}$ 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:

$$K = \frac{(Km)}{1 + 0.0191(T - 25)}$$

5.7.2 Percentage Difference Calculation

For evaluating slope of readings from either a flow cell or a sample cup.

$$\%Difference = \frac{(Highest\ Value - Lowest\ Value)}{(Highest\ Value)} \times 100$$

5.7.3 Convert mm mercury (mmHG) to inches mercury (inHG)

$$mmHG = inHG \times 25.4$$

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.

$$TrueBP = (BP) - \frac{(2.5 \times [Local\ Altitude])}{100}$$

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 mmHG:

$$\text{mmHg} = 30.49 \text{ inHg} \times 25.4 = 774.4 \text{ mmHg}$$

Calculate True BP:

$$\text{TrueBP} = (774.4 \text{ mmHg}) - [2.5 * (544 / 100)] = 774.4 - 13.6 = 760.8 \text{ mmHg}$$

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: _____
Date: _____

Equipment (Make/Model/Serial#): _____
Equipment (Make/Model/Serial#): _____

pH (su) Standard: ± 0.2 standard units				DO (mg/L) Standard: ± 0.3 mg/L of theoretical*			
Initial Calibration		Initial Calibration Verification		IC (Temp: _____)		ICV (Temp: _____)	
Hach SL	Reading	Pine SL	Reading	Saturation (%)	Reading (%)	Theoretical (mg/L)	Reading (mg/L)
pH7	<input type="text"/>	<input type="text"/>	<input type="text"/>	100	<input type="text"/>	<input type="text"/>	<input type="text"/>
pH4	<input type="text"/>	<input type="text"/>	<input type="text"/>				
Continuing Calibration Verification				CCV (Temp: _____)			
Hach SL	Reading	Deviation	Acceptable Variance (Y/N)	Saturation (%)	Reading (%)	Deviation	Acceptable Variance (Y/N)
pH7	<input type="text"/>	<input type="text"/>	<input type="text"/>	100	<input type="text"/>	<input type="text"/>	<input type="text"/>
pH4	<input type="text"/>	<input type="text"/>	<input type="text"/>				
ORP (mV) Standard: NA				Turbidity (ntu) Standard: ± 10% of Standard			
IC (Zobell SL: _____)		ICV (Pine SL: _____)		Initial Calibration			
TCS (Std/Temp)	Reading	TCS (Std/Temp)	Reading	Standard	Reading		
<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>		
CCV (Zobell SL: _____)				Continuing Calibration Verification			
TCS (Std/Temp)	Reading	Deviation	Acceptable Variance (Y/N)	Standard	Reading	Deviation	Acceptable Variance (Y/N)
<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
Conductivity (mS/cm) Standard: ± 5% of standard value				Comments: _____ _____ _____ _____			
IC (YSI SL: _____)		ICV (Pine SL: _____)					
Standard	Reading	Standard	Reading				
<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>				
CCV (YSI SL: _____)							
Standard	Reading	Deviation	Acceptable Variance (Y/N)				
<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>				

Notes: SL solution lit, TCS temperature corrected standard, Std standard, Temp temperature, su standard units, mV millivolts, % percent, mg/L milligrams per liter, ntu nephelometric turbidity units, °C degrees Celsius, m²/cm millimeters per centimeter (temperature corrected), * Theoretical value

Attachment 2
Solubility of Oxygen at Given Temperatures

Field Measurement of Dissolved Oxygen

Solubility of Oxygen in Water at Atmospheric Pressure			
Temperature °C	Oxygen Solubility mg/L	Temperature °C	Oxygen Solubility 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	<input type="checkbox"/> Bailor <input type="checkbox"/> 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 <input type="checkbox"/>

SAMPLE DATA

SAMPLE DATA		<input type="checkbox"/> Bailor <input type="checkbox"/> 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# _____

SW-846, Method 8260B: Data Review Guidelines
DoD QSM v. 4.2 Analysis with NFG Qualifiers
Volatile Organic Compounds by GC/MS

Category	Criteria	Action
<p>Field Samples: Receipt Integrity & Holding Times</p>	<p>Containers received intact with seals unbroken.</p> <p>COC IDs, dates, times, match sample containers.</p> <p>Samples must be received at temp of $\leq 6^{\circ}\text{C}$.</p> <p>Soil samples need to be preserved using pre-tared vial with either sodium bisulfate (low level) or methanol (high level). Note in the data validation report whether the water samples were preserved or not preserved. Note soil collection/preservation technique.</p> <p>Water samples should be received by the lab preserved with HCl to a pH<2, without headspace (bubble-free).</p>	<p>(1) If the cooler temp is $>6^{\circ}\text{C}$ and $<10^{\circ}\text{C}$: Detects = J and Non-detects = UJ</p> <p>(2) If the cooler temp is $<2^{\circ}\text{C}$, but samples are not frozen, do not qualify. If samples are frozen: Detects = J and Non-detects = UJ</p> <p>(3) If cooler temp is $>10^{\circ}\text{C}$: Detects = J and Non-detects = R</p> <p>Do not qualify if the laboratory received the samples on the same day they were collected, it's possible that adequate time did not elapse for the samples to cool.</p> <p>If the sample container was damaged: Detects and Non-detects = R</p> <p>If headspace is $> \frac{1}{4}$" (peas size): Detects = J and Non-detects = R.</p> <p>Include any additional laboratory receipt non-conformance comments in the validation report and discuss potential effects to the data. Apply qualifiers if deemed necessary based on professional judgment.</p>
<p>Holding Times</p>	<p>Preserved waters: 14 days</p> <p>Unpreserved Waters/Soils: 7 days</p> <p>Preserved Soils: 14 days</p>	<p>For preserved waters and soils:</p> <ul style="list-style-type: none"> • analyzed > 14 days: Detects = J and Non-detects = UJ • analyzed > 28 days: Detects = J and Non-detects = R <p>For non-preserved waters or soils:</p> <ul style="list-style-type: none"> • analyzed > 7 days: Detects = J and Non-detects = UJ • analyzed >14 days: Detects = J and Non-detects = R
<p>Method Blank</p>	<p>Minimum one blank per sample batch. The most contaminated MB should be used if multiple MBs were prepared per batch.</p> <p>For the lab to be compliant as specified by DoD QSM, any detects in the method blank should be less than the highest of either $\frac{1}{2}$ the LOQ, 10% regulatory limit, or 10% of sample concentration.</p> <p>All samples prepared in the same batch as the MB are associated for qualification.</p>	<p>For sample detects less than the LOD or less than $1\times$ the associated blank concentration: Detects = UB (at the LOD or blank concentration, whichever is greater) and Non-detects = no qualification</p> <p>For sample detects greater than the LOD and greater than $1\times$ the blank concentration but less than $5\times$ the associated blank concentration: Detects = B, Non-detects = no qualification</p> <p>Apply the same weights, volumes, and dilution factors used for the sample to the associated blank before applying the $1\times$ or $5\times$ rule.</p>

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Volatile Organic Compounds by GC/MS

<p>Trip blanks, equipment blanks, ambient blanks, etc.</p>	<p>Trip blanks, equipment blanks, ambient blank, etc. should be analyte-free.</p> <p>Associated samples are those either collected on the same day, or collected using the same equipment.</p>	<p>For sample detects less than the LOD or less than 1x the associated blank concentration: Detects = UB (at the LOD or blank concentration, whichever is greater) and Non-detects = no qualification</p> <p>For sample detects greater than the LOD and greater than 1x the blank concentration but less than 5x the associated blank concentration: Detects = B, Non-detects = no qualification</p> <p>Apply the same weights, volumes, and dilution factors used for the sample to the associated blank before applying the 1x or 5x rule.</p>
<p>Calibration – Initial (ICAL), Second Source Verification (ICV), & Continuing Calibration Verification (CCV)</p>	<p>Minimum of 5 calibration points. Either the ICAL %RSDs should be $\leq 15\%$ per target analyte, $r \geq 0.995$, or non-linear $r^2 \geq 0.99$ (6 - 7 points required). Resultant curve may not be forced through the origin.</p> <p>The ICAL average RRF should be greater than 0.3 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform and 1,1-dichloroethane.</p> <p>Relative standard deviation (RSD) $\leq 15\%$ or ICAL correlation coefficient (r) should be > 0.995 or $r^2 > 0.99$.</p> <p>ICV - Immediately following ICAL, must be within 20% of true.</p> <p>CCV - Analyzed at beginning of each 12 hr shift. %Ds/% drifts should be $\leq 20\%$.</p>	<p>Analytes in samples corresponding to an out-of-control bracketing ICV/CCV: Detects = J and Non-detects = UJ</p> <p>If $r^2 < 0.99$ or $r < 0.995$ or $RSD \geq 15\%$: Use professional judgment based on location of curve non-linearity versus sample concentrations.</p> <p>If ANY %RSDs/%Ds (absolute value) $> 50\%$, or Recovery $< 50\%$, $> 150\%$ Detects = J and Non-detects = R</p>
<p>GC/MS Tune Criteria</p>	<p>All ICAL and sample associated tunes must meet Method SW-846 criteria.</p>	<p>Qualify all data analyzed following non-compliant tunes using professional judgment.</p>
<p>Surrogates</p>	<p>Surrogate recoveries should be within DoD QSM limits or laboratory control limits for surrogates not specified in QSM.</p> <p>The surrogate concentrations should be constant in all samples and associated batch QC. (If not, note in validation report and use professional judgment for evaluation).</p>	<p>If one or more surrogate %Rs $<$ lower lab limits, but above 20%: Detects = J and Non-detects = UJ</p> <p>If one or more surrogate %R $<$ 20%: Detects = J and Non-detects = R</p> <p>If one or more surrogates $>$ upper lab limit: Detects = J and Non-detects = No qualification</p>

SW-846, Method 8260B: Data Review Guidelines
DoD QSM v. 4.2 Analysis with NFG Qualifiers
Volatile Organic Compounds by GC/MS

<p>Internal Standard Performance</p>	<p>Internal Standard area count should be within 50% to 200% of the mid-point standard in the ICAL.</p> <p>Internal Standard retention time must be within 30 seconds of the mid-point standard from the most recent calibration (CCV).</p>	<p>Qualify all VOC analytes as follows:</p> <p>Sample analysis IS area less than 50% of mid-point standard in the ICAL: Detects = J and Non-detects = R</p> <p>Sample analysis IS area >200% of mid-point standard of the ICAL: Detects = J and Non-detects = none</p> <p>For analytes associated with internal standard retention time > 30 seconds from CCV: Detects which meet spectral ID criteria = J and Non-detects = R</p>
<p>Laboratory Control Sample (LCS)</p>	<p>Minimum one LCS per sample batch.</p> <p>LCS recoveries should be within DoD QSM control limits. Laboratory control limits may not exceed the QSM criteria range. AECOM requires that poor performers must be recovered at 10% of true or greater, see Appendix G of the DoD QSM.</p> <p>DoD Marginal Exceedence (ME) Limits must be used for outlier data evaluation, DoD QSM Section G.2. VOC target lists less than 11 analytes, no ME recoveries are allowed. Full DoD List = 49 targets, 2 LCS ME allowed. LF8 VOC List = 62 targets, 3 LCS ME allowed. No %Rs may be outside the ME limits.</p>	<p>LCS %R < lower limit Detects = J and Non-detects = UJ</p> <p>LCS %R > upper limit: Detects = J and Non-detects = No qualifications</p> <p>LCS %R < ME: Detects = J; Non-detects = R</p> <p>Poor Performers, LCS/LCSD %R<10%, Detects = J, Non-detects = R.</p>
<p>Matrix Spike/Matrix Spike Duplicate (MS/MSD)</p>	<p>Minimum one MS/MSD per 20 investigative samples or per extraction batch.</p> <p>MS/MSD %Rs must be within DoD QSM limits. MS/MSD RPDs must be ≤ 30%.</p> <p>If sample detect concentration is >4× MS/MSD spike concentration, no qualifications required, but mention in report.</p> <p>See the General Comments section below for combining multiple MS.MSD results for qualification per event.</p>	<p>MS and MSD %Rs < lower limit, but >20%: Detects = J and Non-detects = UJ</p> <p>MS and MSD %Rs > upper limit: Detects = J and Non-detects = No qualification</p> <p>MS and/or MSD %R < 20% or <10% for poor performers: Detects = J and Non-detects = R</p> <p>If RPD > 30%: Detects = J; Non-detects = no qualification</p> <p>Note in the report if the MS/MSD samples were reported from a different dilution factor than the parent sample was reported from.</p>
<p>System Performance</p>	<p>Did validation observations include poor agreement between diluted versus undiluted results, were over-dilution practices observed, were RLs supported by the calibration standards range after preparation factors applied, etc?</p> <p>Comment on the general performance of the laboratory, analyst notes, narrative discussions, and any other noted discrepancies or recommendations.</p>	<p>Qualify data using professional judgment when appropriate.</p> <p>Note poor method/laboratory practices, even if qualification not necessary.</p>

SW-846, Method 8260B: Data Review Guidelines
DoD QSM v. 4.2 Analysis with NFG Qualifiers
Volatile Organic Compounds by GC/MS

<p>Misc. Results Qualifying & Reporting</p>	<p>If a dilution was performed and results from both the original and diluted analyses were reported, qualify any (over-range) analyte(s) that required dilution in the original analysis "NP," while leaving the remaining analytes unqualified. Qualify all diluted analyte(s) results that were within range in the original analysis as "NP", while retaining the analyte that required dilution.</p> <p>In most instances, all field QC (like MS/MSDs) are combined and evaluated for data qualification (when >50% results are outliers = all associated data qualified).</p> <p>Field duplicates shall be compared to the RPD criteria when concentrations are >2x the LOQ. Otherwise if <2x LOQ, use \pmLOD for criteria comparison. Duplicate results with one non-detected and one >LOQ, shall cause all results to be qualified. The majority per result cause qualifying. Single outlier, only parent and field duplicate are qualified. Non-detected results do not provide data for determining proportion of outlier results. (For example, if all FD samples replicated a ND result, but one sample pair with detections shows wide variability, all associated event detections must be qualified.)</p>
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Data Review Guidelines
Total Organic Carbon by Walkley-Black

Category	Criteria	Action
Sample Integrity	Samples received intact with seals unbroken. Cooler temp for aqueous and soil samples at <6°C.	<p>If the cooler temp is >6°C and <10°C, Detects = J and Non-detects = UJ</p> <p>If cooler temp is >10°C: Detects = J and Non-detects = R</p> <p>Do not qualify if the laboratory received the samples on the same day they were collected, it's possible that adequate time did not elapse for the samples to cool.</p> <p>If the sample container was damaged: Detects and Non-detects = R</p>
Holding Times	28 Days	<p>If analyzed > 28 and < 85 days: Detects = J and Non-detects = UJ</p> <p>If analyzed ≥ 84 days: Detects = J and Non-detects = R</p>
Method Blank	No analytes detected > LOD	<p>For sample detects less than 5× the associated blank concentration: Detects = B and Non-detects = no qualification</p> <p>Apply the same weights, volumes, and dilution factors used for the sample to the associated blank before applying the above 5× rule.</p>
Laboratory Control Sample (LCS)	<p>Minimum one LCS per preparatory batch.</p> <p>LCS recoveries should be within 85-115%</p>	<p>LCS %Rs < 85% but >30%: Detects = J and Non-detects = UJ</p> <p>LCS %Rs > 115%: Detects = J and Non-detects = No qualifiers</p> <p>LCS %R < 30%: Detects = J and Non-detects = R</p>
Duplicate Sample Analysis	<p>One per preparatory batch .</p> <p>20% RPD for results > 5× LOQ, or ±LOQ for results < 5× LOQ</p>	<p>Qualify affected results: Detects = J and Non-detects = UJ</p>

**Data Review Guidelines
Method 6010C (ICP Metals)**

Category	Criteria	Action
Sample Integrity	Samples received intact with seals unbroken. Cooler temp of <6°C.	<p>For mercury:</p> <p>(1) For soils and waters, if the cooler temp is >6°C and <10°C: Detects = J and Non-detects = UJ</p> <p>(2) If samples are frozen: Detects = J and Non-detects = UJ</p> <p>For all methods (soil and water), if cooler temp is >10°C: Detects = J and Non-detects = R</p> <p>For all metals, if the cooler temp is >6°C and <10°C, note in report but do not qualify.</p> <p>Do not qualify if the laboratory received the samples on the same day they were collected, as there was not enough time allowed for the samples to cool.</p> <p>If the sample container was damaged: Detects and Non-detects = R</p>
	Waters preserved with HNO ₃ to pH<2.	<p>If water sample was not preserved in the field, but was preserved in lab within 2 days of collection, no qualifiers are required.</p> <p>If sample was not preserved or preserved after 2 days past collection: Detects = J and Non-detects = UJ</p>
Blanks (Method, ICB, CCB, equipment blank, etc.)	Minimum one blank per sample batch. Blank should be analyte free. However, for the lab to be compliant as specified by DoD QSM, any detects in the method blank should not be greater than the highest of either ½ the LOQ, 10% regulatory limit, or 10% of sample concentration.	<p>If a blank has a detect (absolute value) above the DL, multiply by 5 and compare to sample results.</p> <p>For sample detects less than 5x the associated positive blank concentration: Detects = B and Non-detects = no qualification</p> <p>For sample detects less than 5x the associated negative blank concentration: Detects = J and Non-detects = UJ</p> <p>Note: When reviewing soils, calculate the effective water DL and use that number to compare to the ICB/CCBs.</p>
Holding Times	6 months.	<p>For metals samples analyzed >6 months and <12 months plus 1 day: Detects = J and Non-detects = UJ</p> <p>For metals samples analyzed beyond 12 months plus one day: Detects = J and Non-detects = R</p>

**Data Review Guidelines
Method 6010C (ICP Metals)**

Category	Criteria	Action
<p>Calibration – Initial (ICAL) & Continuing (ICV/CCV)</p>	<p>For 6010, the ICAL consists of a blank and at least one standard (std).</p> <p>ICV should be within $\pm 10\%$</p> <p>Bracketing CCVs should be within $\pm 10\%$</p>	<p>If $r < 0.995$ or $r^2 < 0.99$: Use professional judgment</p> <p>For samples bracketed by ICV/CCV that are $< \text{limits} > 75\%$: Detects = J and Non-detects = UJ</p> <p>For samples bracketed by ICV/CCV that are $> \text{limits} < 160\%$: Detects = J and Non-detects = UJ</p> <p>For samples bracketed by ICV/CCV that are $< 75\%$: Detects = J and Non-detects = R</p> <p>Samples bracketed by ICV/CCV $< 75\%$ Detects = J Non-detects = R</p> <p>Samples bracketed by ICV/CCV $> 160\%$ Detects = R Non-detects = J</p>
<p>Laboratory Control Sample (LCS)</p>	<p>Minimum one LCS per sample batch.</p> <p>LCS recoveries should be within 80-120%</p>	<p>LCS %Rs $< \text{lower limit}$ but $> 40\%$: Detects = J and Non-detects = UJ</p> <p>LCS %Rs $> \text{upper limit} < 150\%$: Detects = J and Non-detects = No qualifiers</p> <p>LCS %R $< 40\%$: Detects = J and Non-detects = R</p> <p>LCS %R $> 150\%$: All results = R</p>
<p>Matrix Spike/Matrix Spike Duplicate (MS/MSD)</p>	<p>Minimum one MS/MSD per 20 investigative samples.</p> <p>MS/MSD %Rs must be within 80-120%</p> <p>MS/MSD RPDs must be $\leq 20\%$.</p> <p>Qualify all samples of the same matrix, if the reviewer considers the samples sufficiently similar.</p>	<p>MS and MSD %Rs $< \text{lower limit}$, but $> 40\%$: Detects = J and Non-detects = UJ</p> <p>MS and MSD %Rs $> \text{upper limit} < 150\%$: Detects = J and Non-detects = No qualifications</p> <p>MS and/or MSD %R $< 40\%$: Detects = J and Non-detects = R</p> <p>MS and/or MSD %R $> 150\%$: All results = R</p> <p>If sample detect concentration $> 4 \times$ MS/MSD spike concentration, no qualifications required, but mention in report.</p> <p>RPD $> 20\%$: Detects = J and non-detects = UJ</p>

**Data Review Guidelines
Method 6010C (ICP Metals)**

Category	Criteria	Action
Field Duplicate Sample Analysis	<p>Aqueous Samples - 30% RPD for results $>2\times$ LOQ, or \pmLOQ for results $<2\times$ LOQ</p> <p>Solid Samples - 50% RPD for results $>2\times$ LOQ, or \pmLOQ for results $<2\times$ LOQ</p> <p>Qualify the parent and duplicate sample.</p>	Detects = J and non-detects = UJ
Duplicate Sample Analysis	<p>Required if no MS/MSD performed.</p> <p>Aqueous Samples - 20% RPD for results $>5\times$ LOQ, or \pmLOQ for results $<5\times$ LOQ</p> <p>Solid Samples - 20% RPD for results $>5\times$ LOQ, or \pmLOQ for results $<5\times$ LOQ</p> <p>Qualify the parent and duplicate sample.</p>	Detects = J and non-detects = UJ
Interference Check Sample (ICS) – ICP only (ICSA/ICSAB)	Recoveries in the ICSAB should be within $\pm 20\%$. Absolute value of target analytes should be $< LOD$ in the ICSA.	<p>If ICSAB $<80\%$: Detects = J and Non-detects = UJ</p> <p>If ICSAB $>120\%$: Detects = J and Non-detects = no qualification</p> <p>If ICSA $> LOD$: Detects = U and non-detects = no qualification.</p> <p>If a verified trace impurity is documented use professional judgment</p>
Serial Dilution	Results should have a %D $\leq 10\%$, when sample result is $>50\times$ LOQ (as specified in DoD QSM)	<p>If %D $>10\%$ and sample result is $>50\times$ DL: Perform post digestion spike (PDS) (see below)</p> <p>If PDS and MS/MSD are acceptable no qualification. If PDS and MS/MSD not compliant then qualify Detects = J and Non-detects = UJ</p> <p>(Negative interference may be evident when results from diluted analysis are significantly higher than in original sample, therefore, non-detects are also qualified.)</p>
Post-Digestion Spike or Recovery test for GFAA	Post-digestion spike %R must be $>80\%$ or $<120\%$ for ICP	<p>If ICP post-spike %R $<80\%$: Detects = J and Non-detects = UJ</p> <p>If ICP post-spike %R $>120\%$: Detects = J and Non-detects = no qualifications</p> <p>If post-spike or recovery test %R $<40\%$: Detects = J and Non-detects = R</p>
Sample Result Verification (Full Validation only)	Recalculate the result of one ICP result.	If any discrepancies, qualify the data based on professional judgment.
System Performance	Are the data reasonable? Comment on the general performance of the laboratory and any recommendations.	Qualify the concerned data based on professional judgment.

**Data Review Guidelines
Method 6010C (ICP Metals)**

Category	Criteria	Action
Miscellaneous		If a dilution was performed properly and results from both the original and diluted analyses were reported, qualify the analyte that required dilution in the original analysis "NP," while leaving the remaining analytes unqualified. In the diluted results, qualify as "NP" all analytes that were within range in the original analysis while retaining the analyte that required dilution.

Data Review Guidelines
Methane, Ethane, Ethene by RSK 175

Category	Criteria	Action
<p>Field Samples: Receipt Integrity & Holding Times</p>	<p>Containers received intact with seals unbroken.</p> <p>COC IDs, dates, times, match sample containers.</p> <p>Samples must be received at temp of $\leq 6^{\circ}\text{C}$.</p> <p>Water samples should be received by the lab preserved with HCl to a $\text{pH} < 2$, without headspace (bubble-free).</p>	<p>(1) If the cooler temp is $> 6^{\circ}\text{C}$ and $< 10^{\circ}\text{C}$: Detects = J and Non-detects = UJ</p> <p>(2) If the cooler temp is $< 2^{\circ}\text{C}$, but samples are not frozen, do not qualify. If samples are frozen: Detects = J and Non-detects = UJ</p> <p>(3) If cooler temp is $> 10^{\circ}\text{C}$: Detects = J and Non-detects = R</p> <p>Do not qualify if the laboratory received the samples on the same day they were collected, it's possible that adequate time did not elapse for the samples to cool.</p> <p>If the sample container was damaged: Detects and Non-detects = R</p> <p>If headspace is $> \frac{1}{4}$" (peas size): Detects = J and Non-detects = R.</p> <p>Include any additional laboratory receipt non-conformance comments in the validation report and discuss potential effects to the data. Apply qualifiers if deemed necessary based on professional judgment.</p>
<p>Holding Times</p>	<p>Preserved waters: 14 days</p> <p>Unpreserved Waters/Soils: 7 days</p>	<p>For preserved waters:</p> <ul style="list-style-type: none"> • analyzed > 14 days: Detects = J and Non-detects = UJ • analyzed > 28 days: Detects = J and Non-detects = R <p>For non-preserved waters:</p> <ul style="list-style-type: none"> • analyzed > 7 days: Detects = J and Non-detects = UJ • analyzed > 14 days: Detects = J and Non-detects = R
<p>Method Blank</p>	<p>Minimum one blank per preparatory batch. The most contaminated MB should be used if multiple MBs were prepared per batch.</p> <p>Any detects in the method blank should be less than the highest of either $\frac{1}{2}$ the LOQ or 10% of sample concentration.</p> <p>All samples prepared in the same batch as the MB are associated for qualification.</p>	<p>For sample detects less than the LOD or less than $1 \times$ the associated blank concentration: Detects = UB (at the LOD or blank concentration, whichever is greater) and Non-detects = no qualification</p> <p>For sample detects greater than the LOD and greater than $1 \times$ the blank concentration but less than $5 \times$ the associated blank concentration: Detects = B, Non-detects = no qualification</p> <p>Apply the same weights, volumes, and dilution factors used for the sample to the associated blank before applying the $1 \times$ or $5 \times$ rule.</p>

Data Review Guidelines
Methane, Ethane, Ethene by RSK 175

<p>Equipment blanks</p>	<p>Equipment blanks should be analyte-free.</p> <p>Associated samples are those either collected on the same day, or collected using the same equipment.</p>	<p>For sample detects less than the LOD or less than 1x the associated blank concentration: Detects = UB (at the LOD or blank concentration, whichever is greater) and Non-detects = no qualification</p> <p>For sample detects greater than the LOD and greater than 1x the blank concentration but less than 5x the associated blank concentration: Detects = B, Non-detects = no qualification</p> <p>Apply the same weights, volumes, and dilution factors used for the sample to the associated blank before applying the 1x or 5x rule.</p>
<p>Calibration – Initial (ICAL), Second Source Verification (SCV), & Continuing Calibration Verification (CCV)</p>	<p>Minimum of 5 calibration points. Either the ICAL %RSDs should be $\leq 15\%$ per target analyte, $r \geq 0.995$, or non-linear $r^2 \geq 0.99$ (6 - 7 points required). Resultant curve may not be forced through the origin.</p> <p>Relative standard deviation (RSD) $\leq 15\%$ or ICAL correlation coefficient (r) should be >0.995 or $r^2 > 0.99$.</p> <p>SCV - Immediately following ICAL, must be within 20% of true.</p> <p>CCV - Prior to sample analysis, after each 10 field samples, and at the end of the analytical sequence. All project analytes within $\pm \leq 20\%$ of expected value from the ICAL.</p>	<p>Analytes in samples corresponding to an out-of-control bracketing SCV/CCV: Detects = J and Non-detects = UJ</p> <p>If $r^2 < 0.99$ or $r < 0.995$ or $RSD \geq 15\%$: Use professional judgment based on location of curve non-linearity versus sample concentrations.</p> <p>If ANY %RSDs/%Ds (absolute value) $>50\%$, or Recovery $<50\%$, $>150\%$ Detects = J and Non-detects = R</p>
<p>Laboratory Control Sample (LCS)</p>	<p>Minimum one LCS per preparatory batch.</p> <p>LCS recoveries should be within laboratory control limits.</p>	<p>LCS %R $<$ lower limit Detects = J and Non-detects = UJ</p> <p>LCS %R $>$ upper limit: Detects = J and Non-detects = No qualifications</p> <p>LCS %R $<10\%$, Detects = J, Non-detects = R.</p>
<p>Matrix Spike/Matrix Spike Duplicate (MS/MSD)</p>	<p>Minimum one MS/MSD per 20 preparatory batch or one MS/MSD per 20 project samples.</p> <p>MS/MSD %Rs must be within laboratory control limits. MS/MSD RPDs must be $\leq 20\%$.</p> <p>If sample detect concentration is $>4\times$ MS/MSD spike concentration, no qualifications required, but mention in report.</p>	<p>MS and MSD %Rs $<$ lower limit, but $>20\%$: Detects = J and Non-detects = UJ</p> <p>MS and MSD %Rs $>$ upper limit: Detects = J and Non-detects = No qualification</p> <p>MS and/or MSD %R $< 20\%$: Detects = J and Non-detects = R</p> <p>If RPD $> 20\%$: Detects = J; Non-detects = no qualification</p> <p>Note in the report if the MS/MSD samples were reported from a different dilution factor than the parent sample was reported from.</p>

Data Review Guidelines
Methane, Ethane, Ethene by RSK 175

<p>System Performance</p>	<p>Did validation observations include poor agreement between diluted versus undiluted results, were over-dilution practices observed, were RLs supported by the calibration standards range after preparation factors applied, etc?</p> <p>Comment on the general performance of the laboratory, analyst notes, narrative discussions, and any other noted discrepancies or recommendations.</p>	<p>Qualify data using professional judgment when appropriate.</p> <p>Note poor method/laboratory practices, even if qualification not necessary.</p>
<p>Misc. Results Qualifying & Reporting</p>	<p>If a dilution was performed and results from both the original and diluted analyses were reported, qualify any (over-range) analyte(s) that required dilution in the original analysis "NP," while leaving the remaining analytes unqualified. Qualify all diluted analyte(s) results that were within range in the original analysis as "NP", while retaining the analyte that required dilution.</p> <p>In most instances, all field QC (like MS/MSDs) are combined and evaluated for data qualification (when >50% results are outliers = all associated data qualified).</p> <p>Field duplicates shall be compared to the RPD criteria when concentrations are >5x the LOQ. Otherwise if <5x LOQ, use $\pm 2x$ LOQ for criteria comparison. Duplicate results with one non-detected and one >LOQ, shall cause all results to be qualified. The majority per result cause qualifying. Single outlier, only parent and field duplicate are qualified. Non-detected results do not provide data for determining proportion of outlier results. (For example, if all FD samples replicated a ND result, but one sample pair with detections shows wide variability, all associated event detections must be qualified.)</p>	

**Data Review Guidelines
Method 9056A for Anions**

Category	Criteria	Action
Sample Integrity	Samples received intact with seals unbroken. Cooler temp for aqueous and soil samples at <6°C.	<p>For soils and water, if the cooler temp is >6°C and <10°C, Detects = J and Non-detects = UJ</p> <p>For soil and water, if cooler temp is >10°C: Detects = J and Non-detects = R</p> <p>Do not qualify if the laboratory received the samples on the same day they were collected, it's possible that adequate time did not elapse for the samples to cool.</p> <p>If the sample container was damaged: Detects and Non-detects = R</p>
Holding Times	<p>Waters: Bromide (Br⁻), Fluoride (F), Chloride (Cl) and Sulfate (SO₄): 28 days; Nitrate (NO₃), Nitrite (NO₂), and Orthophosphate (PO₄): 48 hrs.</p> <p>Soils: Bromide (Br⁻), Fluoride (F), Chloride (Cl) and Sulfate (SO₄): 28 days; Nitrate (NO₃), Nitrite (NO₂), and Orthophosphate (PO₄): 28 days from collection to extraction and 48 hrs from extraction to analysis</p>	<p>For F, Cl, and SO₄ analyzed > 28 and < 85 days: Detects = J and Non-detects = UJ</p> <p>For F, Cl, and SO₄ analyzed ≥ 84 days: Detects = J and Non-detects = R</p> <p>For NO₃, NO₂, and PO₄ waters analyzed > 48 hours and < 145 hours: Detects = J and Non-detects = UJ</p> <p>For NO₃, NO₂, and PO₄ waters analyzed ≥ 145 hours: Detects = J and Non-detects = R</p> <p>For NO₃, NO₂, and PO₄ soils extracted > 28 and < 85 days, or analyzed > 48 hours and < 145 hours: Detects = J and Non-detects = UJ</p> <p>For NO₃, NO₂, and PO₄ soils extracted ≥ 85 days, or analyzed ≥ 145 hours: Detects = J and Non-detects = UJ</p>
Blanks (method, ICB/CCB)	No analytes detected > ½ 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.	<p>For sample detects less than 5× the associated blank concentration: Detects = B and Non-detects = no qualification</p> <p>Apply the same weights, volumes, and dilution factors used for the sample to the associated blank before applying the above 5x rule.</p>
Calibration – Initial (ICAL), Second Source Verification (ICV), & Continuing Calibration Verification (CCV)	<p>The ICAL should consist of a blank and minimum of 3 stds.</p> <p>The correlation coefficient (r) should be ≥0.995 or r² should be ≥0.99.</p> <p>Bracketing ICV/CCVs should be within lab limits or ±10%.</p>	<p>When r < 0.995 or r² < 0.99: Detects = J and Non-detects = UJ</p> <p>Samples bracketed by ICV/CCVs < limits: Detects = J and Non-detects = UJ</p> <p>Samples bracketed by ICV/CCVs > limits: Detects = J and Non-detects = no qualification</p>

**Data Review Guidelines
Method 9056A for Anions**

Category	Criteria	Action
Laboratory Control Sample (LCS)	<p>Minimum one LCS per sample batch.</p> <p>LCS recoveries should be within QSM limits: 80 - 120%R.</p>	<p>LCS and LCSD %Rs < lower lab limit but >30%: Detects = J and Non-detects = UJ</p> <p>LCS and LCSD %Rs > upper lab limit: Detects = J and Non-detects = No qualifiers</p> <p>LCS and/or LCSD %R < 30%: Detects = J and Non-detects = R</p> <p>LCS/LCSD RPD >15%, all analytical set detections for outlier target = J</p>
MS/MSD	<p>Minimum one MS/MSD per sample batch.</p> <p>MS/MSD recoveries should be within QSM limits: 80 - 120%R.</p> <p>MS/MSD RPD criteria = 15%</p>	<p>MS and MSD %Rs < lower lab limit, but >10%: Detects = J and Non-detects = UJ</p> <p>MS and MSD %Rs > 120%: Detects = J and Non-detects = None</p> <p>MS and/or MSD %R <30%: Detects = J and Non-detects = R</p> <p>If sample detect concentration >4× MS/MSD spike concentration, no qualifications required, but mention in report.</p>
Duplicate Sample Analysis	<p>One per every 10 samples.</p> <p>10% RPD for results > 5× LOQ, or ±LOQ for results < 5× PQL</p>	<p>Qualify affected results: Detects = J and Non-detects = UJ</p>
System Performance	<p>Did validation observations include poor agreement between diluted versus undiluted results, were over-dilution practices observed, were LODs/LOQs supported by the calibration standards range after preparation factors applied, etc?</p> <p>Comment on the general performance of the laboratory, analyst notes, narrative discussions, and any other noted discrepancies or recommendations.</p>	<p>Qualify data using professional judgment when appropriate.</p> <p>Note poor method/laboratory practices, even if qualification not necessary.</p>

**Data Review Guidelines
Method 9056A for Anions**

Category	Criteria	Action
	<p>If a dilution was performed and results from both the original and diluted analyses were reported, qualify any (over-range) analyte(s) that required dilution in the original analysis "NP," while leaving the remaining analytes unqualified. Qualify all diluted analyte(s) results that were within range in the original analysis as "NP", while retaining the analyte that required dilution.</p> <p>In most instances, all field QC (like MS/MSDs) are combined and evaluated for data qualification (when >50% results are outliers = all associated data qualified).</p> <p>Field duplicates shall be compared to a 30% RPD criteria when concentrations are >2x the LOQ. Otherwise if <2x LOQ, use \pmLOD for criteria comparison. Duplicate results with one non-detected and one >LOQ, shall cause results to be qualified. Data qualification will be applied when greater than 50% of the FD results (per method, per target) fail criteria. If only a single outlier is observed, only parent and field duplicate are qualified. Non-detected results do not provide data for determining proportion of outlier results. (For example, if all FD samples replicated a ND result, but one sample pair with detections shows wide variability, all associated event detections must be qualified.)</p>	

APPENDIX C
LABORATORY CONTROL LIMITS, STANDARD OPERATING PROCEDURES
AND LABORATORY CERTIFICATIONS



Minnesota Department of Health
Environmental Laboratory Accreditation Program



Issues accreditation to

State Laboratory ID: 026-999-161

Trimatrix Laboratories, Inc.

5560 Corporate Exchange Court Se

Grand Rapids, MI 49512

for fields of testing listed on the laboratory's accompanying Scope of Certification
in accordance with the provisions in Minnesota Laws and Rules.

Continued accreditation is contingent upon successful on-going compliance with Minnesota Statutes 144.97 to 144.98, 2003 NELAC Standard and applicable Minnesota Rules 4740.2010 to 4740.2120. The laboratory's Scope of Certification cites the specific programs, methods, analytes and matrices (i.e. fields of testing) for which MDH issues this accreditation.

This certificate is valid proof of accreditation only when associated with its accompanying Scope of Certification.

The Scope of Certification and reports of on-site inspections are on file at the Minnesota Department of Health, 601 Robert Street North, Saint Paul, Minnesota. Customers may verify the laboratory's accreditation status in Minnesota by contacting MN-ELAP at (651) 201-5200.

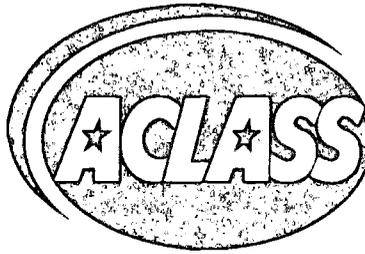
Effective Date: 11/28/2012

Expires: 12/31/2013

A handwritten signature in black ink that reads "Susan R. Wyatt".

Susan R. Wyatt, MN-ELAP Supervisor

Certificate Number: 491715



CERTIFICATE OF ACCREDITATION

ANSI-ASQ National Accreditation Board/AClass
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

TriMatrix Laboratories, Inc.
5560 Corporate Exchange Court, SE
Grand Rapids, MI 49512

has been assessed by AClass
and meets the requirements of

ISO/IEC 17025:2005 and DoD-ELAP

while demonstrating technical competence in the field(s) of

TESTING

Refer to the accompanying Scope(s) of Accreditation for information regarding the types of tests to which this accreditation applies.

ADE-1542

Certificate Number

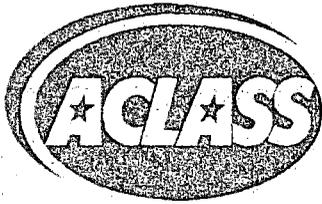
AClass Approval



Certificate Valid: 4/30/2013-04/30/2015
Version No. 003 Issued: 05/30/2013



This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated January 2009).



SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005 & DoD-ELAP

TriMatrix Laboratories, Inc

5560 Corporate Exchange Court, SE, Grand Rapids, MI 49512

Rick Wilburn Phone: 616-975-4500

TESTING

Valid to: April 30, 2015

Certificate Number: ADE - 1542

I. Environmental

MATRIX	SPECIFIC TEST or ANALYTE GROUP**	SPECIFICATION OR STANDARD METHOD (all EPA unless specified)	* KEY EQUIPMENT OR TECHNOLOGY
Water	Metals Digestion	200.2	Block Digestion
Water	Metals Digestion	3010A	Block Digestion
Water	Metals Digestion	3020A	Block Digestion
Solid	Metals Digestion	3050B	Block Digestion
Water	Metals	200.7 / 6010C	ICP
Water	Calcium Hardness (as CaCO ₃)	SM 2340B	ICP
Solid	Metals	6010C	ICP
Water	Total Hardness (as CaCO ₃)	SM 2340B	ICP
Water	Metals	200.8 / 6020A	ICP MS
Solid	Metals	6020A	ICP MS
Water	Mercury	245.1 / 7470A	CVAA
Solid	Mercury	7471B	CVAA



MATRIX	SPECIFIC TEST or ANALYTE GROUP**	SPECIFICATION OR STANDARD METHOD (all EPA unless specified)	* KEY EQUIPMENT OR TECHNOLOGY
Water	Mercury, Low-Level	1631E	CVAF
Water	1,2-Dibromo-3- Chloropropane & 1,2-Dibromoethane	8011	GC-ECD
Water / Solid	Carbonyl Compounds	8315A	HPLC-UV
Water / Solid	Nitroaromatics and Nitramines	8330A	HPLC-UV
Water / Solid	Nitroglycerine and PETN	8332	HPLC-UV
Water / Solid	Chlorinated Herbicides	8151A	GC-ECD
Water	Methoxychlor	608.2	GC-ECD
Water	Organochlorine Pesticides	608 / 8081B	GC-ECD
Solid	Organochlorine Pesticides	8081B	GC-ECD
Water	PCBs	608 / 8082A	GC-ECD
Solid	PCBs	8082A	GC-ECD
Water / Solid	Diesel Range Organics (DRO)	Wisconsin DRO / 8015C	GC-FID
Water / Solid	Oil Range Organics	8015C	GC-FID
Water	Dissolved Gas Analysis	RSK-175	GC-FID
Water / Solid	Nonhalogenated Organics	8015C	GC-FID
Water	Semivolatile Organic Compounds	625 / 8270C	GCMS
Solid	Semivolatile Organic Compounds	8270C	GCMS
Water / Solid	Semivolatile Organic Compounds	8270C SIM	GCMS



MATRIX	SPECIFIC TEST or ANALYTE GROUP**	SPECIFICATION OR STANDARD METHOD (all EPA unless specified)	* KEY EQUIPMENT OR TECHNOLOGY
Solid	Semivolatile Extraction	3545A	Pressurized Fluid Ext
Water	Semivolatile Extraction	3510C	Separatory Funnel Ext
Solid	Semivolatile Extraction	3550C	Ultrasonic Extraction
Water / Solid	Chlorinated Hydrocarbons	8121 / 612	GC-ECD
Water / Solid	Gasoline Range Organics (GRO)	Wisconsin GRO / 8015C	GC-FID
Solid	Volatile Organics	8021B	GC-PID; HECD
Water	Volatiles Organics	601 / 602 / 8021B	GC-PID; HECD
Water	Volatile Organics	524.2 / 624 / 8260B	GCMS
Solid	Volatile Organics	8260B	GCMS
Solid	Volatiles Extraction	5035A	Purge & Trap
Water	Volatiles Extraction	5030B	Purge & Trap/Water
Water / Solid	SPLP	1312	Acetic Acid Leaching
Water / Solid	TCLP	1311	Acetic Acid Leaching
Solid	Ignitability	1020A	Closed-Cup
Solid	Acid Volatile Sulfide and Selected Simultaneously Extractable Metals	821-R-91-100	Distillation/ Spectrophotometric
Solid	Paint Filter Test	9095B	Filtration
Water	HEM Oil and Grease / SGT-HEM Non-Polar Material	1664A / 9070A	Gravimetric



MATRIX	SPECIFIC TEST or ANALYTE GROUP**	SPECIFICATION OR STANDARD METHOD (all EPA unless specified)	* KEY EQUIPMENT OR TECHNOLOGY
Solid	HEM Oil and Grease / SGT-HEM Non-Polar Material	9071B	Gravimetric
Water	Filterable Residue (TDS)	SM 2540C	Gravimetric
Water	Non-Filterable Residue (TSS)	SM 2540D	Gravimetric
Water	Total Residue / Solids (TS)	SM 2540B	Gravimetric
Solid	Total Residue / Solids (TS)	SM 3550C	Gravimetric
Water	Volatile Residue (VS)	SM 2540E	Gravimetric
Solid	Volatile Residue (VS)	SM 2540G	Gravimetric
Water	Settleable Residue / Solids	SM 2540 F	Imhoff cone
Water	Anions	300.0/9056A	Ion Chromatographic
Solid	Anions	9056A	Ion Chromatographic
Water	Bromide	ASTM D1246	ISE
Water / Solid	Fluoride	SM 4500-F C	ISE
Water	BOD and CBOD	SM 5210B	Luminescence
Water	Turbidity	SM 2130B	Nephelometric
Water	Color	SM 2120B	Platinum-Cobalt Color
Water	pH and Corrosivity	SM 4500-H ⁺ B / 9040C	Potentiometric
Solid	pH and Corrosivity	9045D	Potentiometric
Water	Conductivity	SM 2510B / 9050A	Specific Conductance



MATRIX	SPECIFIC TEST or ANALYTE GROUP**	SPECIFICATION OR STANDARD METHOD (all EPA unless specified)	* KEY EQUIPMENT OR TECHNOLOGY
Water / Solid	Ammonia-N	SM 4500-NH ₃ B	Distillation
Water / Solid	Ammonia-N	SM 4500-NH ₃ G	Spectrophotometric
Water	Chloride	SM 4500-Cl E / 9251	Spectrophotometric
Solid	Chromium (VI) Cr ⁻⁶	3060A	Digestion
Water	Chromium (VI) Cr ⁻⁶	SM 3500-Cr B / 7196A	Spectrophotometric
Solid	Chromium (VI) Cr ⁻⁶	7196A	Spectrophotometric
Water	COD	SM 5220D	Spectrophotometric
Water / Solid	Cyanide Available	OIA-1677	Amperometry
Solid	Cyanide	9010C	Distillation
Water	Cyanide	SM 4500-CN C / 9010C	Distillation
Solid	Cyanide Extraction	9013A	Extraction
Solid	Cyanide Amenable	9014	Spectrophotometric
Water	Cyanide Amenable	SM 4500-CN G / 9014	Spectrophotometric
Solid	Cyanide, Total	9014	Spectrophotometric
Water	Cyanide, Total	SM 4500-CN E / 9014	Spectrophotometric
Water	Ferrous Iron	SM 3500-Fe B	Spectrophotometric
Water / Solid	Nitrate-N	SM 4500-NO ₃ F	Spectrophotometric
Water	Nitrate+Nitrite-N	SM 4500-NO ₃ F	Spectrophotometric



MATRIX	SPECIFIC TEST or ANALYTE GROUP**	SPECIFICATION OR STANDARD METHOD (all EPA unless specified)	* KEY EQUIPMENT OR TECHNOLOGY
Water	Nitrite-N	SM 4500-NO ₂ B / NO ₃ F	Spectrophotometric
Water	o-Phosphate	SM 4500-P E	Spectrophotometric
Water / Solid	Phosphorus Total	SM 4500-P E	Spectrophotometric
Water	Silica as SiO ₂	SM 4500-SiO ₂ D	Spectrophotometric
Water	Surfactants (MBAS)	SM 5540C	Spectrophotometric
Water / Solid	TKN	SM 4500-N _{org} D	Spectrophotometric
Solid	Total Phenolics	9065	Spectrophotometric
Water	Total Phenolics	420.4 / 9065	Spectrophotometric
Water	Acidity	SM 2310B	Titrimetric
Water	Calcium Hardness (as CaCO ₃)	SM 2340 C	Titrimetric
Water / Solid	Sulfide	9030B	Distillation
Water	Sulfide	SM 4500-S ₂ D	Spectrophotometric
Water	Sulfide	SM 4500-S ₂ F	Titrimetric
Water / Solid	Sulfide	9034	Titrimetric
Water / Solid	Reactive Sulfide	7.3.4.2	Titrimetric
Water	Sulfite	SM 4500-SO ₃ ²⁻ B	Titrimetric
Water	Total Alkalinity (as CaCO ₃)	SM 2320B	Titrimetric
Water	Total Hardness (as CaCO ₃)	SM 2340C	Titrimetric



MATRIX	SPECIFIC TEST or ANALYTE GROUP**	SPECIFICATION OR STANDARD METHOD (all EPA unless specified)	* KEY EQUIPMENT OR TECHNOLOGY
Water	Sulfate	ASTM D516 / 9038	Turbidimetric
Solid	Total Organic Carbon (TOC)	Lloyd Kahn	Infrared
Water	Total Organic Carbon (TOC)	SM 5310C / 9060A	Oxidation/CO ₂ Det
Solid	Total Organic Carbon (TOC)	WALKLEY BLACK	Titrimetric
Solid	Extractable Organic Halides	9023	Coulometric Titration
Water	Dissolved Organic Carbon	SM 5310C	Titrimetric
Solid	Fractional Organic Carbon	ASTM D2974	Gravimetric
Solid	Grain Size	ASTM D422	Size Exclusion
Water	Total Organic Halides (TOX)	9020B	Coulometric Titration
Solid	% Moisture in Soil and Rock	ASTM D2216	Gravimetric
Solid	Shake Extraction	ASTM D3987	Water Leaching

II. Microbiological

MATRIX	SPECIFIC TEST or ANALYTE GROUP**	SPECIFICATION OR STANDARD METHOD (all EPA unless specified)	* KEY EQUIPMENT OR TECHNOLOGY
Water	Fecal Coliform	SM 9222D	Microbiological
Water	Heterotrophic Bacteria (Std Plate)	SM 9215B	Microbiological
Water	Total Coliform / <i>E. coli</i>	SM 9223B	Microbiological



Notes:

1. * = As Applicable
2. ** = Refer to Accredited Analyte Listing for specific analytes in which the laboratory is accredited.
3. This scope is part of and must be included with the Certificate of Accreditation No. ADE-1542

Karl Brunway

Vice President



Accredited Analytical Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix							
	Aqueous					Solid		
Trace Metals								
Aluminum	200.7	200.8	6010C	6020A			6010C	
Antimony	200.7	200.8	6010C	6020A			6010C	6020A
Arsenic	200.7	200.8	6010C	6020A			6010C	6020A
Barium	200.7	200.8	6010C	6020A			6010C	6020A
Beryllium	200.7	200.8	6010C	6020A			6010C	6020A
Boron	200.7	200.8	6010C	6020A			6010C	6020A
Cadmium	200.7	200.8	6010C	6020A			6010C	6020A
Calcium	200.7		6010C				6010C	
Chromium, total	200.7	200.8	6010C	6020A			6010C	6020A
Chromium VI	SM3500Cr B					7196A		7196A
Cobalt	200.7	200.8	6010C	6020A			6010C	6020A
Copper	200.7	200.8	6010C	6020A			6010C	6020A
Iron	200.7		6010C				6010C	
Lead	200.7	200.8	6010C	6020A			6010C	6020A
Lithium	200.7		6010C				6010C	
Magnesium	200.7		6010C				6010C	
Manganese	200.7	200.8	6010C	6020A			6010C	6020A
Mercury	245.1					7470A		7471B
Mercury (Low Level)						1631E		
Molybdenum	200.7	200.8	6010C	6020A			6010C	6020A
Nickel	200.7	200.8	6010C	6020A			6010C	6020A
Potassium	200.7		6010C				6010C	
Selenium	200.7	200.8	6010C	6020A			6010C	6020A
Silver	200.7	200.8	6010C	6020A			6010C	6020A
Sodium	200.7		6010C				6010C	
Strontium	200.7	200.8	6010C	6020A			6010C	
Thallium	200.7	200.8	6010C	6020A			6010C	6020A
Tin	200.7	200.8	6010C	6020A			6010C	6020A
Titanium	200.7		6010C				6010C	
Vanadium	200.7	200.8	6010C	6020A			6010C	6020A
Zinc	200.7	200.8	6010C	6020A			6010C	6020A
Demands								
TOC	SM5310C	9060A					Walkley Black	Lloyd Kahn
COD			SM5220D					
DOC-Dissolved Organic Carbon							SM5310C	

Accredited Analytes/Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix							
	Aqueous				Solid			
BOD and CBOD	SM5210B							
Misc Analytes								
Total Alkalinity (as CaCO ₃)	SM2320B							
Calcium Hardness as CaCO ₃	SM2340B							
Total Hardness as CaCO ₃	SM2340B	SM2340C						
% Moisture in Soil and Rock						ASTM D2216		
Total Residue/Solids (TS)	SM2540B					3550C		
Filterable Residue (TDS)		SM2540C						
Ignitability						1020A		
Non-Filterable Residue (TSS)	SM2540D							
Volatile Residue (VS)		SM2540E				SM2540G		
Settleable Residue	SM2540F							
pH			9040C	SM4500H ⁺ B		9045D		
Sulfide	SM4500S ²⁻ D	SM4500S ²⁻ F	9034			9034		
Total Cyanide	SM4500CN E	9014				9014		
Cyanide, Amenable	SM4500CN G	9014				9014		
Ammonia	SM4500NH ₃ G					SM4500NH ₃ G		
Conductivity	SM2510B	9050A						
Nitrogen, Total Kjeldahl (TKN)	SM4500N _{org} D					SM4500N _{org} D		
Total Phenolics	420.4	9065				9065		
Total Organic Halides (TOX)	9020B							
Bromide	ASTM D1246		300.0	9056A				
Chloride	SM4500Cl E	9251	300.0	9056A		9056A		
Fluoride	SM4500F C		300.0	9056A		9056A		
Nitrate as N	SM4500NO ₃ F		300.0	9056A		9056A	SM4500NO ₃ F	
Nitrite as N	SM4500NO ₂ B	SM4500NO ₃ F	300.0	9056A		9056A		
Nitrate + Nitrite as N	SM4500NO ₃ F		300.0	9056A		9056A		
ortho-phosphate	SM4500P E							
Total Phosphorus	SM4500P E					SM4500P E		
Silica as SiO ₂	SM4500SiO ₂ D							
Sulfate	ASTM D516	9038	300.0	9056A		9056A		
Surfactants - MBAS	SM5540C							
Fecal Coliform	SM9222D							
Heterotrophic Bacteria (Std Plate)	SM9215B							
Total Coliform	SM9223B							

Accredited Analytes/Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix							
	Aqueous						Solid	
Turbidity		SM2130B						
EOX-Extractable Organic Halides							9023	
FOC-Fractional Organic Carbon							ASTM D2974	
Petroleum Hydrocarbons								
SGT-HEM; Non-Polar Material	1664A	9070A					9071B	
HEM; Oil and Grease	1664A	9070A					9071B	
Gasoline Range Organics (GRO)	8015C	Wisconsin GRO					8015C	Wisconsin GRO
Diesel Range Organics (DRO)	8015C	Wisconsin DRO					8015C	Wisconsin DRO
Oil Range Organics (ORO)	8015C						8015C	
VOCs								
Acetone		624	8260B					8260B
Acetonitrile		624	8260B					8260B
Acrolein		624	8260B					8260B
Acrylonitrile		624	8260B					8260B
Benzene	524.2	624	8260B		602	8021B	8021B	8260B
Bromobenzene	524.2		8260B					8260B
Bromochloromethane	524.2		8260B					8260B
Bromodichloromethane	524.2	624	8260B	601		8021B	8021B	8260B
Bromoform	524.2	624	8260B	601		8021B	8021B	8260B
Bromomethane	524.2	624	8260B	601		8021B	8021B	8260B
2-Butanone (MEK)		624	8260B					8260B
n-Butylbenzene	524.2		8260B					8260B
sec-Butylbenzene	524.2		8260B					8260B
tert-Butylbenzene	524.2		8260B					8260B
Carbon disulfide		624	8260B					8260B
Carbon Tetrachloride	524.2	624	8260B	601		8021B	8021B	8260B
Chlorobenzene	524.2	624	8260B	601	602	8021B	8021B	8260B
Chlorodibromomethane	524.2	624	8260B	601		8021B	8021B	8260B
Chloroethane	524.2	624	8260B	601		8021B	8021B	8260B
2-Chloroethylvinylether		624	8260B	601		8021B	8021B	8260B
Chloroform	524.2	624	8260B	601		8021B	8021B	8260B
Chloromethane	524.2	624	8260B	601		8021B	8021B	8260B
2-Chlorotoluene	524.2		8260B					8260B
4-Chlorotoluene	524.2		8260B					8260B
1,2-Dibromo-3-chloropropane (DBCP)		624	8260B					8260B

Accredited Analytes/Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix							
	Aqueous				Solid			
1,2-Dibromoethane (EDB)		624	8260B					8260B
Dibromomethane	524.2	624	8260B					8260B
1,2-Dichlorobenzene	524.2	624	8260B	601	602	8021B	8021B	8260B
1,3-Dichlorobenzene	524.2	624	8260B	601	602	8021B	8021B	8260B
1,4-Dichlorobenzene	524.2	624	8260B	601	602	8021B	8021B	8260B
Dichlorodifluoromethane	524.2	624	8260B	601		8021B	8021B	8260B
1,1-Dichloroethane	524.2	624	8260B	601		8021B	8021B	8260B
1,2-Dichloroethane	524.2	624	8260B	601		8021B	8021B	8260B
1,1-Dichloroethene	524.2	624	8260B	601		8021B	8021B	8260B
<i>cis</i> -1,2-Dichloroethene	524.2	624	8260B	601		8021B	8021B	8260B
<i>trans</i> -1,2-Dichloroethene	524.2	624	8260B	601		8021B	8021B	8260B
Dichloromethane (Methylene Chloride)	524.2	624	8260B	601		8021B	8021B	8260B
1,2-Dichloropropane	524.2	624	8260B	601		8021B	8021B	8260B
1,3-Dichloropropane	524.2		8260B					8260B
2,2-Dichloropropane	524.2		8260B					8260B
1,1-Dichloropropene	524.2		8260B					8260B
<i>cis</i> -1,3-Dichloropropene	524.2	624	8260B	601		8021B	8021B	8260B
<i>trans</i> -1,3-Dichloropropene	524.2	624	8260B	601		8021B	8021B	8260B
Di-isopropylether (DIPE)			8260B					8260B
Ethylbenzene	524.2	624	8260B		602	8021B	8021B	8260B
Ethanol			8260B			8015C	8015C	8260B
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)			8260B					8260B
Hexachlorobutadiene	524.2	624	8260B					8260B
2-Hexanone		624	8260B					8260B
Isopropylbenzene	524.2	624	8260B					8260B
4-Isopropyltoluene	524.2		8260B					8260B
4-Methyl-2-pentanone (MIBK)		624	8260B					8260B
Methyl-tert-butylether (MTBE)	524.2	624	8260B					8260B
Naphthalene	524.2	624	8260B					8260B
n-Propylbenzene	524.2		8260B					8260B
Styrene	524.2	624	8260B					8260B
tert-amylmethylether (TAME)			8260B					8260B
1,1,1,2-Tetrachloroethane	524.2	624	8260B					8260B
1,1,1,2,2-Tetrachloroethane	524.2	624	8260B	601		8021B	8021B	8260B
Tetrachloroethene	524.2	624	8260B	601		8021B	8021B	8260B
Toluene	524.2	624	8260B		602	8021B	8021B	8260B
1,2,3-Trichlorobenzene	524.2		8260B					8260B

Accredited Analytes Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix								
	Aqueous						Solid		
1,2,4-Trichlorobenzene	524.2	624	8260B					8260B	
1,1,1-Trichloroethane	524.2	624	8260B	601		8021B	8021B	8260B	
1,1,2-Trichloroethane	524.2	624	8260B	601		8021B	8021B	8260B	
Trichloroethene	524.2	624	8260B	601		8021B	8021B	8260B	
Trichlorofluoromethane (Freon 11)	524.2	624	8260B	601		8021B	8021B	8260B	
1,2,3-Trichloropropane	524.2	624	8260B					8260B	
1,2,4-Trimethylbenzene	524.2		8260B					8260B	
1,3,5-Trimethylbenzene	524.2		8260B					8260B	
Vinyl acetate		624	8260B					8260B	
Vinyl chloride	524.2	624	8260B	601		8021B	8021B	8260B	
o-Xylene		624	8260B		602			8260B	
m+p-Xylene		624	8260B		602			8260B	
Xylenes, total	524.2	624	8260B		602	8021B	8021B	8260B	
SVOCs - Base/Neutrals/Acids									
Acenaphthene	8270C SIM	625	8270C				8270C	8270C SIM	
Acenaphthylene	8270C SIM	625	8270C				8270C	8270C SIM	
Aniline		625	8270C				8270C		
Anthracene	8270C SIM	625	8270C				8270C	8270C SIM	
Benzo(a)anthracene	8270C SIM	625	8270C				8270C	8270C SIM	
Benzo(b)fluoranthene	8270C SIM	625	8270C				8270C	8270C SIM	
Benzo(k)fluoranthene	8270C SIM	625	8270C				8270C	8270C SIM	
Benzo(g,h,i)perylene	8270C SIM	625	8270C				8270C	8270C SIM	
Benzo(a)pyrene	8270C SIM	625	8270C				8270C	8270C SIM	
Benzidine		625	8270C				8270C		
Benzoic acid		625	8270C				8270C		
Benzyl alcohol		625	8270C				8270C		
4-Bromophenyl-phenylether		625	8270C				8270C		
Butyl benzyl phthalate		625	8270C				8270C		
Carbazole			8270C				8270C		
4-Chloroaniline		625	8270C				8270C		
bis(2-Chloroethoxy)methane		625	8270C				8270C		
bis(2-Chloroethyl)ether		625	8270C				8270C		
bis(2-Chloroisopropyl) ether		625	8270C				8270C		
4-Chloro-3-methylphenol		625	8270C				8270C		
2-Chloronaphthalene		625	8270C	612		8121	8270C	8121	
4-Chlorophenyl-phenylether		625	8270C				8270C		

Accredited Analytes/Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix							
	Aqueous				Solid			
2-Chlorophenol		625	8270C				8270C	
Chrysene	8270C SIM	625	8270C				8270C	8270C SIM
Dibenzo(a,h)anthracene	8270C SIM	625	8270C				8270C	8270C SIM
Dibenzofuran		625	8270C				8270C	
Di-n-butylphthalate		625	8270C				8270C	
1,2-Dichlorobenzene		625	8270C	612		8121	8270C	8121
1,3-Dichlorobenzene		625	8270C	612		8121	8270C	8121
1,4-Dichlorobenzene		625	8270C	612		8121	8270C	8121
3,3'-Dichlorobenzidine		625	8270C				8270C	
2,4-Dichlorophenol		625	8270C				8270C	
2,6-Dichlorophenol		625	8270C				8270C	
Diethyl phthalate		625	8270C				8270C	
2,4-Dimethylphenol		625	8270C				8270C	
Dimethylphthalate		625	8270C				8270C	
Diphenylamine			8270C				8270C	
2,4-Dinitrophenol		625	8270C				8270C	
2,4-Dinitrotoluene		625	8270C				8270C	
2,6-Dinitrotoluene		625	8270C				8270C	
Di-n-octylphthalate		625	8270C				8270C	
Dinoseb			8270C				8270C	
1,4-Dioxane			8270C					
bis(2-ethylhexyl) phthalate		625	8270C				8270C	
Fluoranthene	8270C SIM	625	8270C				8270C	8270C SIM
Fluorene	8270C SIM	625	8270C				8270C	8270C SIM
Hexachlorobenzene		625	8270C	612		8121	8270C	8121
Hexachlorobutadiene		625	8270C	612		8121	8270C	8121
Hexachlorocyclopentadiene		625	8270C	612		8121	8270C	8121
Hexachloroethane		625	8270C	612		8121	8270C	8121
Indeno(1,2,3, cd)pyrene	8270C SIM	625	8270C				8270C	8270C SIM
Isophorone		625	8270C				8270C	
2-Methyl-4,6-Dinitrophenol		625	8270C				8270C	
2-Methylphenol		625	8270C				8270C	
4-Methylphenol (and/or 3-Methylphenol)		625	8270C				8270C	
2-Methylnaphthalene	8270C SIM	625	8270C				8270C	8270C SIM
Naphthalene	8270C SIM	625	8270C				8270C	8270C SIM
2-Nitroaniline		625	8270C				8270C	
3-Nitroaniline		625	8270C				8270C	

Accredited Analytes/Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix							
	Aqueous					Solid		
		625	8270C					
4-Nitroaniline		625	8270C				8270C	
Nitrobenzene		625	8270C				8270C	
2-Nitrophenol		625	8270C				8270C	
4-Nitrophenol		625	8270C				8270C	
4-nitroquinoline-1-oxide			8270C				8270C	
N-Nitrosodiethylamine		625	8270C				8270C	
N-Nitrosodimethylamine		625	8270C				8270C	
N-Nitrosodiphenylamine		625	8270C				8270C	
N-Nitroso-di-n-propylamine		625	8270C				8270C	
Pentachlorobenzene		625	8270C				8270C	
Pentachlorophenol		625	8270C				8270C	
Phenanthrene	8270C SIM	625	8270C				8270C	8270C SIM
Phenol		625	8270C				8270C	
Pyrene	8270C SIM	625	8270C				8270C	8270C SIM
Pyridine		625	8270C				8270C	
1,2,4,5-Tetrachlorobenzene		625	8270C				8270C	
2,3,4,6-Tetrachlorophenol		625	8270C				8270C	
o-Toluidine		625	8270C				8270C	
1,2,4-Trichlorobenzene		625	8270C	612		8121	8270C	8121
2,4,5-Trichlorophenol		625	8270C				8270C	
2,4,6-Trichlorophenol		625	8270C				8270C	
Nitroaromatic and Nitramines								
4-Amino-2,6-dinitrotoluene	8330A						8330A	
2-Amino-4,6-dinitrotoluene	8330A						8330A	
1,3-Dinitrobenzene	8330A						8330A	
2,4-Dinitrotoluene	8330A						8330A	
2,6-Dinitrotoluene	8330A						8330A	
HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)	8330A						8330A	
Nitrobenzene	8330A						8330A	
Nitroglycerin	8330A	8332					8330A	8332
2-Nitrotoluene	8330A						8330A	
3-Nitrotoluene	8330A						8330A	
4-Nitrotoluene	8330A						8330A	
Pentaerythritol tetranitrate	8330A	8332					8330A	8332
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	8330A						8330A	
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	8330A						8330A	

Accredited Analytes/Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix								
	Aqueous				Solid				
1,3,5-Trinitrobenzene	8330A							8330A	
2,4,6-Trinitrotoluene	8330A							8330A	
Pesticides									
Aldrin	608	8081B						8081B	
alpha-BHC	608	8081B						8081B	
beta-BHC	608	8081B						8081B	
delta-BHC	608	8081B						8081B	
gamma-BHC (Lindane)	608	8081B						8081B	
alpha-Chlordane	608	8081B						8081B	
gamma-Chlordane	608	8081B						8081B	
Chlordane (technical)	608	8081B						8081B	
DDD (4,4)	608	8081B						8081B	
DDE (4,4)	608	8081B						8081B	
DDT (4,4)	608	8081B						8081B	
Dieldrin	608	8081B						8081B	
Endosulfan I	608	8081B						8081B	
Endosulfan II	608	8081B						8081B	
Endosulfan sulfate	608	8081B						8081B	
Endrin	608	8081B						8081B	
Endrin aldehyde	608	8081B						8081B	
Endrin ketone	608	8081B						8081B	
Heptachlor	608	8081B						8081B	
Heptachlor Epoxide (beta)	608	8081B						8081B	
Methoxychlor	608.2	8081B						8081B	
Toxaphene (total)	608	8081B						8081B	
Organophosphorus Pesticides									
Dimethoate		8270C						8270C	
Dichlorvos		8270C						8270C	
Disulfoton		8270C						8270C	
Parathion, ethyl		8270C						8270C	
Parathion, methyl		8270C						8270C	
Phorate		8270C						8270C	
Sulfotepp		8270C						8270C	
Herbicides									

Accredited Analytes Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix								
	Aqueous				Solid				
2,4,5-T	8151A							8151A	
2,4,5-TP (Silvex)	8151A							8151A	
2,4-D	8151A							8151A	
2,4-DB	8151A							8151A	
Dalapon	8151A							8151A	
Dicamba	8151A							8151A	
Dichloroprop	8151A							8151A	
Dinoseb	8151A							8151A	
MCPA	8151A							8151A	
MCPP	8151A							8151A	
Pentachlorophenol	8151A							8151A	
Picloram	8151A							8151A	
PCBs									
Aroclor 1016	608	8082A						8082A	
Aroclor 1221	608	8082A						8082A	
Aroclor 1232	608	8082A						8082A	
Aroclor 1242	608	8082A						8082A	
Aroclor 1248	608	8082A						8082A	
Aroclor 1254	608	8082A						8082A	
Aroclor 1260	608	8082A						8082A	
Misc. Analytes -Additional									
Iron, Ferrous	SM 3500Fe B								
Cyanide, Available	OIA-1677							OIA-1677	
Acidity	SM 2310 B								
Sulfite	SM 4500SO ₃ ²⁻ B								
Paint Filter Liquids Test								9095B	
Color	SM 2120 B								
Acid Volatile Sulfides (AVS/SEM)								EPA-821-R-91-100	
Grain Size								ASTM D422-63	
Reactive Sulfide	7.3.4.2							7.4.3.2	
PCBs - Additional Aroclors									
Aroclor 1262	608	8082A						8082A	
Aroclor 1268	608	8082A						8082A	

Accredited Analytes/Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix					
	Aqueous			Solid		
Misc. Organics						
Ethane	RSK-175					
Ethylene	RSK-175					
Methane	RSK-175					
Additional Compounds						
Volatiles						
1,2-dibromo-3-chloropropane	8011					
1,2-dibromoethane	8011					
1,2,3-Trimethylbenzene			8260B			8260B
1,4-dioxane			8260B			8260B
1-chlorohexane			8260B			8260B
sec-butanol		8015C	8260B		8015C	8260B
2-chloro-1,3-butadiene (Chloroprene)			8260B			8260B
2-methylnaphthalene			8260B			8260B
2-nitropropane			8260B			8260B
allyl chloride			8260B			8260B
cyclohexane			8260B			8260B
ETBE			8260B			8260B
ethanol		8015C			8015C	
ethyl acetate			8260B			8260B
ethyl ether			8260B			8260B
ethyl methacrylate			8260B			8260B
hexachloroethane			8260B			8260B
hexane			8260B			8260B
iodomethane			8260B			8260B
isobutanol		8015C	8260B		8015C	8260B
isopropanol		8015C	8260B		8015C	8260B
methacrylonitrile			8260B			8260B
methanol		8015C			8015C	
methyl acetate			8260B			8260B
methyl methacrylate			8260B			8260B
methylcyclohexane			8260B			8260B
n-butanol		8015C	8260B		8015C	8260B
n-butyl acetate			8260B			8260B
n-propanol		8015C	8260B		8015C	8260B

Accredited Analytes/Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix								
	Aqueous					Solid			
propionitrile				8260B				8260B	
t-butanol		8015C		8260B			8015C	8260B	
tetrahydrofuran				8260B				8260B	
trans -1,4,dichloro-2-butene				8260B				8260B	
trichlorotrifluoromethane				8260B				8260B	
SVOCs - Base/Neutrals/Acids									
1,1'-Biphenyl				8270C			8270C		
1,2-Bis(2-chloroethoxy)ethane				8270C			8270C		
1,2-Diphenylhydrazine				8270C			8270C		
1,3 Dinitrobenzene				8270C			8270C		
1,4-Naphthoquinone				8270C			8270C		
1,4-Phenylenediamine				8270C			8270C		
1-Methylnaphthalene				8270C			8270C		
1-Naphthylamine				8270C			8270C		
1-Nitrosopyrrolidine				8270C			8270C		
2-Acetylaminofluorene				8270C			8270C		
2-Chloroaniline				8270C			8270C		
2-Naphthylamine				8270C			8270C		
2-Picoline				8270C			8270C		
3,3'-Dimethylbenzidine				8270C			8270C		
3-Methylchloanthrene				8270C			8270C		
4-Aminobiphenyl				8270C			8270C		
5-Nitro-o-toluidine				8270C			8270C		
7,12-Dimethylbenz(a)anthracene				8270C			8270C		
a,a-Dimethylphenethylamine				8270C			8270C		
Acetophenone				8270C			8270C		
Aramite				8270C			8270C		
Atrazine				8270C			8270C		
Benzaldehyde				8270C			8270C		
Bis(2-ethylhexyl) adipate				8270C			8270C		
Caprolactam				8270C			8270C		
Chlorobenzilate				8270C			8270C		
Diallate				8270C			8270C		
Dicyclohexyl Phthalate				8270C			8270C		
Ethyl Methacrylate				8270C			8270C		
Ethyl Methansulfonate				8270C			8270C		

Accredited Analytes/Methods (by matrix)

TriMatrix Laboratoires, Inc.

Grand Rapids, MI

Analyte	Matrix							
	Aqueous				Solid			
Famphur			8270C				8270C	
Hexachlorophene			8270C				8270C	
Hexachloropropene			8270C				8270C	
Isodrin			8270C				8270C	
Isosafrole			8270C				8270C	
Kepon			8270C				8270C	
Methapyrilene			8270C				8270C	
Methyl Methacrylate			8270C				8270C	
Methyl Methanesulfonate			8270C				8270C	
N-Nitroso-di-n-butylamine			8270C				8270C	
N-Nitrosomethylethylamine			8270C				8270C	
N-Nitrosomorpholine			8270C				8270C	
N-Nitrosopiperidine			8270C				8270C	
o,o,o-Triethylphosphorothioate			8270C				8270C	
p-Dimethylaminoazobenzene			8270C				8270C	
Phenacetin			8270C				8270C	
Pentachloroethane			8270C				8270C	
Pentachloronitrobenzene			8270C				8270C	
Pronamide			8270C				8270C	
Safrole			8270C				8270C	
Thionazin			8270C				8270C	
1,3,5-Trinitrobenzene			8270C				8270C	
Carbonyls								
Formaldehyde	8315A						8315A	
Acetaldehyde	8315A						8315A	
Propanal	8315A						8315A	
Crotonaldehyde	8315A						8315A	
Butanal	8315A						8315A	
Pentanal	8315A						8315A	
Cyclohexanone	8315A						8315A	
m-Tolualdehyde	8315A						8315A	
Hexanal	8315A						8315A	
Heptanal	8315A						8315A	
Octanal	8315A						8315A	
Nonanal	8315A						8315A	
Decanal	8315A						8315A	



*Environmental Laboratory Certification Program
Scope of Certification
Certified Minnesota Environmental Laboratories*

**THIS LISTING OF CERTIFIED FIELDS OF TESTING MUST BE
ACCOMPANIED BY CERTIFICATE NUMBER: 491715**

State Laboratory ID: 026-999-161

EPA Lab Code: MI00005

Expiration Date: 12/31/2013

Issue Date: 11/28/2012

Trimatrix Laboratories, Inc.
5560 Corporate Exchange Court Se
Grand Rapids, MI 49512

Clean Water Program

ASTM D516-90

Preparation Techniques: N/A

<u>Program</u>	<u>Method</u>	<u>Analyte</u>	<u>Matrix</u>	<u>Primary</u>	<u>SOP</u>
CWP	ASTM D516-90	Sulfate	NPW	FL	

EPA 160.4

Preparation Techniques: N/A

<u>Program</u>	<u>Method</u>	<u>Analyte</u>	<u>Matrix</u>	<u>Primary</u>	<u>SOP</u>
CWP	EPA 160.4	Residue-volatile	NPW	FL	

EPA 1631E

Preparation Techniques: Digestion, hotplate or HotBlock;

<u>Program</u>	<u>Method</u>	<u>Analyte</u>	<u>Matrix</u>	<u>Primary</u>	<u>SOP</u>
CWP	EPA 1631E	Mercury	NPW	FL	

EPA 1664A (HEM)

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1664A (HEM)	Oil & Grease	NPW	FL	

EPA 1664A (SGT-HEM)

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1664A (SGT-HEM)	Oil & Grease	NPW	FL	

EPA 200.7

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.7	Aluminum	NPW	FL	
CWP	EPA 200.7	Arsenic	NPW	FL	
CWP	EPA 200.7	Barium	NPW	FL	
CWP	EPA 200.7	Beryllium	NPW	FL	
CWP	EPA 200.7	Boron	NPW	FL	
CWP	EPA 200.7	Cadmium	NPW	FL	
CWP	EPA 200.7	Calcium	NPW	FL	
CWP	EPA 200.7	Cobalt	NPW	FL	
CWP	EPA 200.7	Copper	NPW	FL	
CWP	EPA 200.7	Iron	NPW	FL	
CWP	EPA 200.7	Lead	NPW	FL	
CWP	EPA 200.7	Magnesium	NPW	FL	
CWP	EPA 200.7	Manganese	NPW	FL	
CWP	EPA 200.7	Molybdenum	NPW	FL	
CWP	EPA 200.7	Nickel	NPW	FL	
CWP	EPA 200.7	Potassium	NPW	FL	
CWP	EPA 200.7	Selenium	NPW	FL	
CWP	EPA 200.7	Silver	NPW	FL	
CWP	EPA 200.7	Sodium	NPW	FL	
CWP	EPA 200.7	Thallium	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.7	Tin	NPW	FL	
CWP	EPA 200.7	Total chromium	NPW	FL	
CWP	EPA 200.7	Vanadium	NPW	FL	
CWP	EPA 200.7	Zinc	NPW	FL	

EPA 200.8

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.8	Antimony	NPW	FL	
CWP	EPA 200.8	Arsenic	NPW	FL	
CWP	EPA 200.8	Barium	NPW	FL	
CWP	EPA 200.8	Beryllium	NPW	FL	
CWP	EPA 200.8	Cadmium	NPW	FL	
CWP	EPA 200.8	Cobalt	NPW	FL	
CWP	EPA 200.8	Copper	NPW	FL	
CWP	EPA 200.8	Lead	NPW	FL	
CWP	EPA 200.8	Manganese	NPW	FL	
CWP	EPA 200.8	Molybdenum	NPW	FL	
CWP	EPA 200.8	Nickel	NPW	FL	
CWP	EPA 200.8	Selenium	NPW	FL	
CWP	EPA 200.8	Silver	NPW	FL	
CWP	EPA 200.8	Thallium	NPW	FL	
CWP	EPA 200.8	Total chromium	NPW	FL	
CWP	EPA 200.8	Vanadium	NPW	FL	
CWP	EPA 200.8	Zinc	NPW	FL	

EPA 245.1

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 245.1	Mercury	NPW	FL	

EPA 300.0

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 300.0	Chloride	NPW	FL	
CWP	EPA 300.0	Fluoride	NPW	FL	
CWP	EPA 300.0	Nitrate as N	NPW	FL	
CWP	EPA 300.0	Nitrite as N	NPW	FL	
CWP	EPA 300.0	Sulfate	NPW	FL	

EPA 351.2

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 351.2	Kjeldahl nitrogen - total	NPW	FL	

EPA 420.4

Preparation Techniques: Distillation, macro;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 420.4	Total Phenolics	NPW	FL	

EPA 608

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 608	4,4'-DDD	NPW	FL	
CWP	EPA 608	4,4'-DDE	NPW	FL	
CWP	EPA 608	4,4'-DDT	NPW	FL	
CWP	EPA 608	Aldrin	NPW	FL	
CWP	EPA 608	alpha-BHC (alpha-Hexachlorocyclohexane)	NPW	FL	
CWP	EPA 608	Aroclor-1016 (PCB-1016)	NPW	FL	
CWP	EPA 608	Aroclor-1221 (PCB-1221)	NPW	FL	
CWP	EPA 608	Aroclor-1232 (PCB-1232)	NPW	FL	
CWP	EPA 608	Aroclor-1242 (PCB-1242)	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 608	Aroclor-1248 (PCB-1248)	NPW	FL	
WP	EPA 608	Aroclor-1254 (PCB-1254)	NPW	FL	
CWP	EPA 608	Aroclor-1260 (PCB-1260)	NPW	FL	
CWP	EPA 608	beta-BHC (beta-Hexachlorocyclohexane)	NPW	FL	
CWP	EPA 608	Chlordane (tech.)	NPW	FL	
CWP	EPA 608	delta-BHC	NPW	FL	
CWP	EPA 608	Dieldrin	NPW	FL	
CWP	EPA 608	Endosulfan I	NPW	FL	
CWP	EPA 608	Endosulfan II	NPW	FL	
CWP	EPA 608	Endosulfan sulfate	NPW	FL	
CWP	EPA 608	Endrin	NPW	FL	
CWP	EPA 608	Endrin aldehyde	NPW	EL	
CWP	EPA 608	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	NPW	FL	
CWP	EPA 608	Heptachlor	NPW	FL	
CWP	EPA 608	Heptachlor epoxide	NPW	FL	
CWP	EPA 608	Toxaphene (Chlorinated camphene)	NPW	FL	

PA 624

Preparation Techniques: Purge and trap;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 624	1,1,1-Trichloroethane	NPW	FL	
CWP	EPA 624	1,1,2,2-Tetrachloroethane	NPW	FL	
CWP	EPA 624	1,1,2-Trichloroethane	NPW	FL	
CWP	EPA 624	1,1-Dichloroethane	NPW	FL	
CWP	EPA 624	1,1-Dichloroethylene	NPW	FL	
CWP	EPA 624	1,2-Dichlorobenzene	NPW	FL	
CWP	EPA 624	1,2-Dichloroethane (Ethylene dichloride)	NPW	FL	
CWP	EPA 624	1,2-Dichloropropane	NPW	FL	
CWP	EPA 624	1,3-Dichlorobenzene	NPW	FL	
CWP	EPA 624	1,4-Dichlorobenzene	NPW	FL	
CWP	EPA 624	2-Chloroethyl vinyl ether	NPW	FL	
CWP	EPA 624	Acrolein (Propenal)	NPW	FL	
CWP	EPA 624	Acrylonitrile	NPW	FL	
WP	EPA 624	Benzene	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 624	Bromodichloromethane	NPW	FL	
CWP	EPA 624	Bromoform	NPW	FL	
CWP	EPA 624	Carbon tetrachloride	NPW	FL	
CWP	EPA 624	Chlorobenzene	NPW	FL	
CWP	EPA 624	Chlorodibromomethane	NPW	FL	
CWP	EPA 624	Chloroethane (Ethyl chloride)	NPW	FL	
CWP	EPA 624	Chloroform	NPW	FL	
CWP	EPA 624	cis-1,3-Dichloropropene	NPW	FL	
CWP	EPA 624	Ethylbenzene	NPW	FL	
CWP	EPA 624	Methyl bromide (Bromomethane)	NPW	FL	
CWP	EPA 624	Methyl chloride (Chloromethane)	NPW	FL	
CWP	EPA 624	Methylene chloride (Dichloromethane)	NPW	FL	
CWP	EPA 624	Tetrachloroethylene (Perchloroethylene)	NPW	FL	
CWP	EPA 624	Toluene	NPW	FL	
CWP	EPA 624	trans-1,2-Dichloroethylene	NPW	FL	
CWP	EPA 624	trans-1,3-Dichloropropylene	NPW	FL	
CWP	EPA 624	Trichloroethene (Trichloroethylene)	NPW	FL	
CWP	EPA 624	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	NPW	FL	
CWP	EPA 624	Vinyl chloride	NPW	FL	

EPA 625

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 625	1,2,4-Trichlorobenzene	NPW	FL	
CWP	EPA 625	2,4,6-Trichlorophenol	NPW	FL	
CWP	EPA 625	2,4-Dichlorophenol	NPW	FL	
CWP	EPA 625	2,4-Dimethylphenol	NPW	FL	
CWP	EPA 625	2,4-Dinitrophenol	NPW	FL	
CWP	EPA 625	2,4-Dinitrotoluene (2,4-DNT)	NPW	FL	
CWP	EPA 625	2,6-Dinitrotoluene (2,6-DNT)	NPW	FL	
CWP	EPA 625	2-Chloronaphthalene	NPW	FL	
CWP	EPA 625	2-Chlorophenol	NPW	FL	
CWP	EPA 625	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	NPW	FL	
CWP	EPA 625	2-Nitrophenol	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 625	3,3'-Dichlorobenzidine	NPW	FL	
WP	EPA 625	4-Bromophenyl phenyl ether	NPW	FL	
CWP	EPA 625	4-Chloro-3-methylphenol	NPW	FL	
CWP	EPA 625	4-Chlorophenyl phenylether	NPW	FL	
CWP	EPA 625	4-Nitrophenol	NPW	FL	
CWP	EPA 625	Acenaphthene	NPW	FL	
CWP	EPA 625	Acenaphthylene	NPW	FL	
CWP	EPA 625	Anthracene	NPW	FL	
CWP	EPA 625	Benzidine	NPW	FL	
CWP	EPA 625	Benzo(a)anthracene	NPW	FL	
CWP	EPA 625	Benzo(a)pyrene	NPW	FL	
CWP	EPA 625	Benzo(g,h,i)perylene	NPW	FL	
CWP	EPA 625	Benzo(k)fluoranthene	NPW	FL	
CWP	EPA 625	Benzo[b]fluoranthene	NPW	FL	
CWP	EPA 625	bis(2-Chloroethoxy)methane	NPW	FL	
CWP	EPA 625	bis(2-Chloroethyl) ether	NPW	FL	
CWP	EPA 625	bis(2-Chloroisopropyl) ether	NPW	FL	
CWP	EPA 625	Butyl benzyl phthalate	NPW	FL	
WP	EPA 625	Chrysene	NPW	FL	
CWP	EPA 625	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	NPW	FL	
CWP	EPA 625	Di-n-butyl phthalate	NPW	FL	
CWP	EPA 625	Di-n-octyl phthalate	NPW	FL	
CWP	EPA 625	Dibenz(a,h) anthracene	NPW	FL	
CWP	EPA 625	Diethyl phthalate	NPW	FL	
CWP	EPA 625	Dimethyl phthalate	NPW	FL	
CWP	EPA 625	Fluoranthene	NPW	FL	
CWP	EPA 625	Fluorene	NPW	FL	
CWP	EPA 625	Hexachlorobenzene	NPW	FL	
CWP	EPA 625	Hexachlorobutadiene	NPW	FL	
CWP	EPA 625	Hexachlorocyclopentadiene	NPW	FL	
CWP	EPA 625	Hexachloroethane	NPW	FL	
CWP	EPA 625	Indeno(1,2,3-cd) pyrene	NPW	FL	
CWP	EPA 625	Isophorone	NPW	FL	
CWP	EPA 625	n-Nitrosodi-n-propylamine	NPW	FL	
CWP	EPA 625	n-Nitrosodimethylamine	NPW	FL	
CWP	EPA 625	n-Nitrosodiphenylamine	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 625	Naphthalene	NPW	FL	
CWP	EPA 625	Nitrobenzene	NPW	FL	
CWP	EPA 625	Pentachlorophenol	NPW	FL	
CWP	EPA 625	Phenanthrene	NPW	FL	
CWP	EPA 625	Phenol	NPW	FL	
CWP	EPA 625	Pyrene	NPW	FL	

OIA 1677

Preparation Techniques: Distillation, MIDI; Distillation, macro;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	OIA 1677	Free cyanide	NPW	FL	

SM 2120 B-93

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2120 B-93	Color	NPW	FL	

SM 2130 B-94

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2130 B-94	Turbidity	NPW	FL	

SM 2310 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2310 B-97	Acidity, as CaCO ₃	NPW	FL	

SM 2320 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2320 B-97	Alkalinity as CaCO3	NPW	FL	

SM 2340 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2340 B-97	Total hardness as CaCO3	NPW	FL	

SM 2340 C-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2340 C-97	Total hardness as CaCO3	NPW	FL	

SM 2510 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2510 B-97	Conductivity	NPW	FL	

SM 2540 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 B-97	Residue-total	NPW	FL	

SM 2540 C-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 C-97	Residue-filterable (TDS)	NPW	FL	

SM 2540 D-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 D-97	Residue-nonfilterable (TSS)	NPW	FL	

SM 2540 F-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 F-97	Residue-settleable	NPW	FL	

SM 3500-Cr B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 3500-Cr B-97	Chromium VI	NPW	FL	

SM 3500-Fe B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 3500-Fe B-97	Iron	NPW	FL	

SM 4500-Cl⁻E-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-Cl ⁻ E-97	Chloride	NPW	FL	

SM 4500-CN⁻E-97

Preparation Techniques: Distillation, MIDI; Distillation, macro;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-CN ⁻ E-97	Total Cyanide	NPW	FL	

SM 4500-F⁻C-97

Preparation Techniques: Distillation, MIDI; Distillation, macro;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-F ⁻ C-97	Fluoride	NPW	FL	

SM 4500-H⁺B-96

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-H ⁺ B-96	pH	NPW	FL	

SM 4500-NH₃G-97

Preparation Techniques: Distillation, MIDI; Distillation, macro;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-NH ₃ G-97	Ammonia as N	NPW	FL	

SM 4500-NO₂⁻B-93

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-NO ₂ ⁻ B-93	Nitrite as N	NPW	FL	

SM 4500-NO₃⁻F-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-NO ₃ ⁻ F-97	Nitrate as N	NPW	FL	
NP	SM 4500-NO ₃ ⁻ F-97	Nitrate-nitrite	NPW	FL	
CWP	SM 4500-NO ₃ ⁻ F-97	Nitrite as N	NPW	FL	

SM 4500-P E-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-P E-97	Orthophosphate as P	NPW	FL	
CWP	SM 4500-P E-97	Total Phosphorus	NPW	FL	

SM 4500-S2⁻ D-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-S2 ⁻ D-97	Sulfide	NPW	FL	

SM 4500-S2⁻ F-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-S2 ⁻ F-97	Sulfide	NPW	FL	

SM 4500-SiO₂ C-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-SiO ₂ C-97	Silica-dissolved	NPW	FL	

SM 4500-SO₃⁻ B-96

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-SO ₃ ⁻ B-96	Sulfite-SO ₃	NPW	FL	

SM 5210 B

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 5210 B	Carbonaceous BOD, CBOD	NPW	FL	

SM 5220 D-97

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 5220 D-97	Chemical oxygen demand	NPW	FL	

SM 5310 C-96

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 5310 C-96	Total Organic Carbon	NPW	FL	

SM 5540 C-93

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 5540 C-93	Surfactants - MBAS	NPW	FL	

Resource Conservation Recovery Program

EPA 1020A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1020A	Ignitability	SCM	FL	

EPA 6010B

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CRP	EPA 6010B	Aluminum	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010B	Aluminum	SCM	FL	
RCRP	EPA 6010B	Arsenic	NPW	FL	
RCRP	EPA 6010B	Arsenic	SCM	FL	
RCRP	EPA 6010B	Barium	SCM	FL	
RCRP	EPA 6010B	Barium	NPW	FL	
RCRP	EPA 6010B	Beryllium	NPW	FL	
RCRP	EPA 6010B	Beryllium	SCM	FL	
RCRP	EPA 6010B	Boron	SCM	FL	
RCRP	EPA 6010B	Boron	NPW	FL	
RCRP	EPA 6010B	Cadmium	SCM	FL	
RCRP	EPA 6010B	Cadmium	NPW	FL	
RCRP	EPA 6010B	Calcium	NPW	FL	
RCRP	EPA 6010B	Calcium	SCM	FL	
RCRP	EPA 6010B	Chromium	SCM	FL	
RCRP	EPA 6010B	Cobalt	NPW	FL	
RCRP	EPA 6010B	Cobalt	SCM	FL	
RCRP	EPA 6010B	Copper	SCM	FL	
RCRP	EPA 6010B	Copper	NPW	FL	
RCRP	EPA 6010B	Iron	SCM	FL	
RCRP	EPA 6010B	Iron	NPW	FL	
RCRP	EPA 6010B	Lead	NPW	FL	
RCRP	EPA 6010B	Lead	SCM	FL	
RCRP	EPA 6010B	Lithium	SCM	FL	
RCRP	EPA 6010B	Lithium	NPW	FL	
RCRP	EPA 6010B	Magnesium	NPW	FL	
RCRP	EPA 6010B	Magnesium	SCM	FL	
RCRP	EPA 6010B	Manganese	SCM	FL	
RCRP	EPA 6010B	Manganese	NPW	FL	
RCRP	EPA 6010B	Molybdenum	SCM	FL	
RCRP	EPA 6010B	Molybdenum	NPW	FL	
RCRP	EPA 6010B	Nickel	SCM	FL	
RCRP	EPA 6010B	Nickel	NPW	FL	
RCRP	EPA 6010B	Potassium	NPW	FL	
RCRP	EPA 6010B	Potassium	SCM	FL	
RCRP	EPA 6010B	Selenium	SCM	FL	
RCRP	EPA 6010B	Selenium	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010B	Silver	NPW	FL	
CRP	EPA 6010B	Silver	SCM	FL	
RCRP	EPA 6010B	Sodium	NPW	FL	
RCRP	EPA 6010B	Sodium	SCM	FL	
RCRP	EPA 6010B	Strontium	SCM	FL	
RCRP	EPA 6010B	Strontium	NPW	FL	
RCRP	EPA 6010B	Thallium	SCM	FL	
RCRP	EPA 6010B	Thallium	NPW	FL	
RCRP	EPA 6010B	Tin	NPW	FL	
RCRP	EPA 6010B	Tin	SCM	FL	
RCRP	EPA 6010B	Titanium	NPW	FL	
RCRP	EPA 6010B	Titanium	SCM	FL	
RCRP	EPA 6010B	Total chromium	NPW	FL	
RCRP	EPA 6010B	Vanadium	SCM	FL	
RCRP	EPA 6010B	Vanadium	NPW	FL	
RCRP	EPA 6010B	Zinc	NPW	FL	
RCRP	EPA 6010B	Zinc	SCM	FL	

EPA 6020

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020	Antimony	NPW	FL	
RCRP	EPA 6020	Antimony	SCM	FL	
RCRP	EPA 6020	Arsenic	SCM	FL	
RCRP	EPA 6020	Arsenic	NPW	FL	
RCRP	EPA 6020	Barium	SCM	FL	
RCRP	EPA 6020	Barium	NPW	FL	
RCRP	EPA 6020	Beryllium	NPW	FL	
RCRP	EPA 6020	Beryllium	SCM	FL	
RCRP	EPA 6020	Cadmium	SCM	FL	
RCRP	EPA 6020	Cadmium	NPW	FL	
RCRP	EPA 6020	Chromium	SCM	FL	
RCRP	EPA 6020	Cobalt	SCM	FL	
CRP	EPA 6020	Cobalt	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020	Copper	NPW	FL	
RCRP	EPA 6020	Copper	SCM	FL	
RCRP	EPA 6020	Lead	NPW	FL	
RCRP	EPA 6020	Lead	SCM	FL	
RCRP	EPA 6020	Manganese	NPW	FL	
RCRP	EPA 6020	Manganese	SCM	FL	
RCRP	EPA 6020	Nickel	NPW	FL	
RCRP	EPA 6020	Nickel	SCM	FL	
RCRP	EPA 6020	Silver	NPW	FL	
RCRP	EPA 6020	Silver	SCM	FL	
RCRP	EPA 6020	Thallium	NPW	FL	
RCRP	EPA 6020	Thallium	SCM	FL	
RCRP	EPA 6020	Total chromium	NPW	FL	
RCRP	EPA 6020	Zinc	NPW	FL	
RCRP	EPA 6020	Zinc	SCM	FL	

EPA 7196A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 7196A	Chromium VI	NPW	FL	

EPA 7470A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 7470A	Mercury	NPW	FL	

EPA 7471A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 7471A	Mercury	SCM	FL	

EPA 8015B

Preparation Techniques: Purge and trap; Extraction, EPA 1312 SPLP, zero headspace (ZHE); Extraction, separatory funnel liquid-liquid (LLE); Extraction, ultrasonic; Extraction, pressurized fluid (PFE); Extraction, EPA 1311 TCLP, zero headspace (ZHE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8015B	Ethanol	NPW	FL	
RCRP	EPA 8015B	Ethanol	SCM	FL	
RCRP	EPA 8015B	Isobutyl alcohol (2-Methyl-1-propanol)	NPW	FL	
RCRP	EPA 8015B	Isobutyl alcohol (2-Methyl-1-propanol)	SCM	FL	
RCRP	EPA 8015B	Isopropyl alcohol (2-Propanol, Isopropanol)	SCM	FL	
RCRP	EPA 8015B	Isopropyl alcohol (2-Propanol, Isopropanol)	NPW	FL	
RCRP	EPA 8015B	Methanol	NPW	FL	
RCRP	EPA 8015B	Methanol	SCM	FL	
RCRP	EPA 8015B	n-Butyl alcohol (1-Butanol, n-Butanol)	SCM	FL	
RCRP	EPA 8015B	n-Butyl alcohol (1-Butanol, n-Butanol)	NPW	FL	
RCRP	EPA 8015B	tert-Butyl alcohol	SCM	FL	

EPA 8081A

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Extraction, EPA 1312 SPLP, non-volatiles; Extraction, separatory funnel liquid-liquid (LLE); Extraction, ultrasonic; Extraction, pressurized fluid (PFE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8081A	4,4'-DDD	SCM	FL	
RCRP	EPA 8081A	4,4'-DDD	NPW	FL	
RCRP	EPA 8081A	4,4'-DDE	NPW	FL	
RCRP	EPA 8081A	4,4'-DDE	SCM	FL	
RCRP	EPA 8081A	4,4'-DDT	SCM	FL	
RCRP	EPA 8081A	4,4'-DDT	NPW	FL	
RCRP	EPA 8081A	Aldrin	SCM	FL	
RCRP	EPA 8081A	Aldrin	NPW	FL	
RCRP	EPA 8081A	alpha-BHC (alpha-Hexachlorocyclohexane)	SCM	FL	
RCRP	EPA 8081A	alpha-BHC (alpha-Hexachlorocyclohexane)	NPW	FL	
RCRP	EPA 8081A	alpha-Chlordane	NPW	FL	
RCRP	EPA 8081A	alpha-Chlordane	SCM	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8081A	beta-BHC (beta-Hexachlorocyclohexane)	SCM	FL	
RCRP	EPA 8081A	beta-BHC (beta-Hexachlorocyclohexane)	NPW	FL	
RCRP	EPA 8081A	Chlordane (tech.)	NPW	FL	
RCRP	EPA 8081A	Chlordane (tech.)	SCM	FL	
RCRP	EPA 8081A	delta-BHC	SCM	FL	
RCRP	EPA 8081A	delta-BHC	NPW	FL	
RCRP	EPA 8081A	Dieldrin	NPW	FL	
RCRP	EPA 8081A	Dieldrin	SCM	FL	
RCRP	EPA 8081A	Endosulfan I	SCM	FL	
RCRP	EPA 8081A	Endosulfan I	NPW	FL	
RCRP	EPA 8081A	Endosulfan II	NPW	FL	
RCRP	EPA 8081A	Endosulfan II	SCM	FL	
RCRP	EPA 8081A	Endosulfan sulfate	SCM	FL	
RCRP	EPA 8081A	Endosulfan sulfate	NPW	FL	
RCRP	EPA 8081A	Endrin	NPW	FL	
RCRP	EPA 8081A	Endrin	SCM	FL	
RCRP	EPA 8081A	Endrin aldehyde	NPW	FL	
RCRP	EPA 8081A	Endrin aldehyde	SCM	FL	
RCRP	EPA 8081A	Endrin ketone	NPW	FL	
RCRP	EPA 8081A	Endrin ketone	SCM	FL	
RCRP	EPA 8081A	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	SCM	FL	
RCRP	EPA 8081A	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	NPW	FL	
RCRP	EPA 8081A	gamma-Chlordane	SCM	FL	
RCRP	EPA 8081A	gamma-Chlordane	NPW	FL	
RCRP	EPA 8081A	Heptachlor	SCM	FL	
RCRP	EPA 8081A	Heptachlor	NPW	FL	
RCRP	EPA 8081A	Heptachlor epoxide	NPW	FL	
RCRP	EPA 8081A	Heptachlor epoxide	SCM	FL	
RCRP	EPA 8081A	Methoxychlor	SCM	FL	
RCRP	EPA 8081A	Methoxychlor	NPW	FL	
RCRP	EPA 8081A	Toxaphene (Chlorinated camphene)	SCM	FL	
RCRP	EPA 8081A	Toxaphene (Chlorinated camphene)	NPW	FL	

EPA 8082

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Extraction, EPA 1312 SPLP, non-volatiles; Extraction, separatory funnel liquid-liquid (LLE); Extraction, pressurized fluid (PFE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8082	Aroclor-1016 (PCB-1016)	NPW	FL	
RCRP	EPA 8082	Aroclor-1016 (PCB-1016)	SCM	FL	
RCRP	EPA 8082	Aroclor-1221 (PCB-1221)	SCM	FL	
RCRP	EPA 8082	Aroclor-1221 (PCB-1221)	NPW	FL	
RCRP	EPA 8082	Aroclor-1232 (PCB-1232)	SCM	FL	
RCRP	EPA 8082	Aroclor-1232 (PCB-1232)	NPW	FL	
RCRP	EPA 8082	Aroclor-1242 (PCB-1242)	NPW	FL	
RCRP	EPA 8082	Aroclor-1242 (PCB-1242)	SCM	FL	
RCRP	EPA 8082	Aroclor-1248 (PCB-1248)	SCM	FL	
RCRP	EPA 8082	Aroclor-1248 (PCB-1248)	NPW	FL	
RCRP	EPA 8082	Aroclor-1254 (PCB-1254)	NPW	FL	
RCRP	EPA 8082	Aroclor-1254 (PCB-1254)	SCM	FL	
RCRP	EPA 8082	Aroclor-1260 (PCB-1260)	NPW	FL	
RCRP	EPA 8082	Aroclor-1260 (PCB-1260)	SCM	FL	
RCRP	EPA 8082	PCBs	SCM	FL	

EPA 8151A

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Extraction, EPA 1312 SPLP, non-volatiles; Extraction, separatory funnel liquid-liquid (LLE); Extraction, pressurized fluid (PFE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8151A	2,4,5-T	NPW	FL	
RCRP	EPA 8151A	2,4,5-T	SCM	FL	
RCRP	EPA 8151A	2,4-D	NPW	FL	
RCRP	EPA 8151A	2,4-D	SCM	FL	
RCRP	EPA 8151A	2,4-DB	NPW	FL	
RCRP	EPA 8151A	2,4-DB	SCM	FL	
RCRP	EPA 8151A	Dalapon	NPW	FL	
RCRP	EPA 8151A	Dalapon	SCM	FL	
RCRP	EPA 8151A	Dicamba	SCM	FL	
RCRP	EPA 8151A	Dicamba	NPW	FL	
RCRP	EPA 8151A	Dichloroprop (Dichloroprop)	NPW	FL	
RCRP	EPA 8151A	Dichloroprop (Dichloroprop)	SCM	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8151A	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	SCM	FL	
RCRP	EPA 8151A	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	NPW	FL	
RCRP	EPA 8151A	MCPA	NPW	FL	
RCRP	EPA 8151A	MCPA	SCM	FL	
RCRP	EPA 8151A	MCPP	NPW	FL	
RCRP	EPA 8151A	MCPP	SCM	FL	
RCRP	EPA 8151A	Silvex (2,4,5-TP)	SCM	FL	
RCRP	EPA 8151A	Silvex (2,4,5-TP)	NPW	FL	

EPA 8260B

Preparation Techniques: Purge and trap; Extraction, EPA 1312 SPLP, zero headspace (ZHE); Extraction, EPA 1311 TCLP, zero headspace (ZHE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	1,1,1,2-Tetrachloroethane	NPW	FL	
RCRP	EPA 8260B	1,1,1,2-Tetrachloroethane	SCM	FL	
RCRP	EPA 8260B	1,1,1-Trichloroethane	NPW	FL	
RCRP	EPA 8260B	1,1,1-Trichloroethane	SCM	FL	
RCRP	EPA 8260B	1,1,2,2-Tetrachloroethane	SCM	FL	
RCRP	EPA 8260B	1,1,2,2-Tetrachloroethane	NPW	FL	
RCRP	EPA 8260B	1,1,2-Trichloroethane	SCM	FL	
RCRP	EPA 8260B	1,1,2-Trichloroethane	NPW	FL	
RCRP	EPA 8260B	1,1-Dichloroethane	SCM	FL	
RCRP	EPA 8260B	1,1-Dichloroethane	NPW	FL	
RCRP	EPA 8260B	1,1-Dichloroethylene	NPW	FL	
RCRP	EPA 8260B	1,1-Dichloroethylene	SCM	FL	
RCRP	EPA 8260B	1,1-Dichloropropene	NPW	FL	
RCRP	EPA 8260B	1,1-Dichloropropene	SCM	FL	
RCRP	EPA 8260B	1,2,3-Trichlorobenzene	SCM	FL	
RCRP	EPA 8260B	1,2,3-Trichlorobenzene	NPW	FL	
RCRP	EPA 8260B	1,2,3-Trichloropropane	SCM	FL	
RCRP	EPA 8260B	1,2,3-Trichloropropane	NPW	FL	
RCRP	EPA 8260B	1,2,4-Trichlorobenzene	SCM	FL	
RCRP	EPA 8260B	1,2,4-Trichlorobenzene	NPW	FL	
RCRP	EPA 8260B	1,2,4-Trimethylbenzene	SCM	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	1,2,4-Trimethylbenzene	NPW	FL	
RCRP	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)	NPW	FL	
RCRP	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)	SCM	FL	
RCRP	EPA 8260B	1,2-Dibromoethane (EDB, Ethylene dibromide)	NPW	FL	
RCRP	EPA 8260B	1,2-Dibromoethane (EDB, Ethylene dibromide)	SCM	FL	
RCRP	EPA 8260B	1,2-Dichlorobenzene	SCM	FL	
RCRP	EPA 8260B	1,2-Dichlorobenzene	NPW	FL	
RCRP	EPA 8260B	1,2-Dichloroethane (Ethylene dichloride)	SCM	FL	
RCRP	EPA 8260B	1,2-Dichloroethane (Ethylene dichloride)	NPW	FL	
RCRP	EPA 8260B	1,2-Dichloropropane	NPW	FL	
RCRP	EPA 8260B	1,2-Dichloropropane	SCM	FL	
RCRP	EPA 8260B	1,3,5-Trimethylbenzene	NPW	FL	
RCRP	EPA 8260B	1,3,5-Trimethylbenzene	SCM	FL	
RCRP	EPA 8260B	1,3-Dichlorobenzene	NPW	FL	
RCRP	EPA 8260B	1,3-Dichlorobenzene	SCM	FL	
RCRP	EPA 8260B	1,3-Dichloropropane	SCM	FL	
RCRP	EPA 8260B	1,3-Dichloropropane	NPW	FL	
RCRP	EPA 8260B	1,4-Dichlorobenzene	NPW	FL	
RCRP	EPA 8260B	1,4-Dichlorobenzene	SCM	FL	
RCRP	EPA 8260B	1,4-Dioxane (1,4- Diethyleneoxide)	NPW	FL	
RCRP	EPA 8260B	1,4-Dioxane (1,4- Diethyleneoxide)	SCM	FL	
RCRP	EPA 8260B	1-Chlorohexane	NPW	FL	
RCRP	EPA 8260B	1-Chlorohexane	SCM	FL	
RCRP	EPA 8260B	2,2-Dichloropropane	NPW	FL	
RCRP	EPA 8260B	2,2-Dichloropropane	SCM	FL	
RCRP	EPA 8260B	2-Butanone (Methyl ethyl ketone, MEK)	SCM	FL	
RCRP	EPA 8260B	2-Butanone (Methyl ethyl ketone, MEK)	NPW	FL	
RCRP	EPA 8260B	2-Chloroethyl vinyl ether	SCM	FL	
RCRP	EPA 8260B	2-Chloroethyl vinyl ether	NPW	FL	
RCRP	EPA 8260B	2-Chlorotoluene	SCM	FL	
RCRP	EPA 8260B	2-Chlorotoluene	NPW	FL	
RCRP	EPA 8260B	2-Hexanone	NPW	FL	
RCRP	EPA 8260B	2-Hexanone	SCM	FL	
RCRP	EPA 8260B	2-Nitropropane	NPW	FL	
RCRP	EPA 8260B	4-Chlorotoluene	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	4-Chlorotoluene	SCM	FL	
RCRP	EPA 8260B	4-Isopropyltoluene (p-Cymene)	SCM	FL	
RCRP	EPA 8260B	4-Isopropyltoluene (p-Cymene)	NPW	FL	
RCRP	EPA 8260B	4-Methyl-2-pentanone (MIBK)	SCM	FL	
RCRP	EPA 8260B	4-Methyl-2-pentanone (MIBK)	NPW	FL	
RCRP	EPA 8260B	Acetone	NPW	FL	
RCRP	EPA 8260B	Acetone	SCM	FL	
RCRP	EPA 8260B	Acetonitrile	NPW	FL	
RCRP	EPA 8260B	Acetonitrile	SCM	FL	
RCRP	EPA 8260B	Acrolein (Propenal)	SCM	FL	
RCRP	EPA 8260B	Acrolein (Propenal)	NPW	FL	
RCRP	EPA 8260B	Acrylonitrile	NPW	FL	
RCRP	EPA 8260B	Acrylonitrile	SCM	FL	
RCRP	EPA 8260B	Allyl chloride (3-Chloropropene)	NPW	FL	
RCRP	EPA 8260B	Allyl chloride (3-Chloropropene)	SCM	FL	
RCRP	EPA 8260B	Benzene	SCM	FL	
RCRP	EPA 8260B	Benzene	NPW	FL	
RCRP	EPA 8260B	Bromobenzene	SCM	FL	
RCRP	EPA 8260B	Bromobenzene	NPW	FL	
RCRP	EPA 8260B	Bromochloromethane	SCM	FL	
RCRP	EPA 8260B	Bromochloromethane	NPW	FL	
RCRP	EPA 8260B	Bromodichloromethane	NPW	FL	
RCRP	EPA 8260B	Bromodichloromethane	SCM	FL	
RCRP	EPA 8260B	Bromoform	SCM	FL	
RCRP	EPA 8260B	Bromoform	NPW	FL	
RCRP	EPA 8260B	Carbon disulfide	NPW	FL	
RCRP	EPA 8260B	Carbon disulfide	SCM	FL	
RCRP	EPA 8260B	Carbon tetrachloride	NPW	FL	
RCRP	EPA 8260B	Carbon tetrachloride	SCM	FL	
RCRP	EPA 8260B	Chlorobenzene	NPW	FL	
RCRP	EPA 8260B	Chlorobenzene	SCM	FL	
RCRP	EPA 8260B	Chlorodibromomethane	NPW	FL	
RCRP	EPA 8260B	Chlorodibromomethane	SCM	FL	
RCRP	EPA 8260B	Chloroethane (Ethyl chloride)	SCM	FL	
RCRP	EPA 8260B	Chloroethane (Ethyl chloride)	NPW	FL	
RCRP	EPA 8260B	Chloroform	SCM	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	Chloroform	NPW	FL	
RCRP	EPA 8260B	Chloroprene (2-Chloro-1,3-butadiene)	NPW	FL	
RCRP	EPA 8260B	Chloroprene (2-Chloro-1,3-butadiene)	SCM	FL	
RCRP	EPA 8260B	cis-1,2-Dichloroethylene	SCM	FL	
RCRP	EPA 8260B	cis-1,2-Dichloroethylene	NPW	FL	
RCRP	EPA 8260B	cis-1,3-Dichloropropene	SCM	FL	
RCRP	EPA 8260B	cis-1,3-Dichloropropene	NPW	FL	
RCRP	EPA 8260B	Dibromomethane (Methylene bromide)	SCM	FL	
RCRP	EPA 8260B	Dibromomethane (Methylene bromide)	NPW	FL	
RCRP	EPA 8260B	Dichlorodifluoromethane (Freon-12)	NPW	FL	
RCRP	EPA 8260B	Dichlorodifluoromethane (Freon-12)	SCM	FL	
RCRP	EPA 8260B	Diethyl ether	SCM	FL	
RCRP	EPA 8260B	Diethyl ether	NPW	FL	
RCRP	EPA 8260B	Ethanol	SCM	FL	
RCRP	EPA 8260B	Ethanol	NPW	FL	
RCRP	EPA 8260B	Ethyl acetate	NPW	FL	
RCRP	EPA 8260B	Ethyl acetate	SCM	FL	
RCRP	EPA 8260B	Ethyl methacrylate	SCM	FL	
RCRP	EPA 8260B	Ethyl methacrylate	NPW	FL	
RCRP	EPA 8260B	Ethylbenzene	SCM	FL	
RCRP	EPA 8260B	Ethylbenzene	NPW	FL	
RCRP	EPA 8260B	Hexachlorobutadiene	NPW	FL	
RCRP	EPA 8260B	Hexachlorobutadiene	SCM	FL	
RCRP	EPA 8260B	Hexachloroethane	NPW	FL	
RCRP	EPA 8260B	Hexachloroethane	SCM	FL	
RCRP	EPA 8260B	Iodomethane (Methyl iodide)	SCM	FL	
RCRP	EPA 8260B	Iodomethane (Methyl iodide)	NPW	FL	
RCRP	EPA 8260B	Isobutyl alcohol (2-Methyl-1-propanol)	SCM	FL	
RCRP	EPA 8260B	Isobutyl alcohol (2-Methyl-1-propanol)	NPW	FL	
RCRP	EPA 8260B	Isopropyl alcohol (2-Propanol, Isopropanol)	NPW	FL	
RCRP	EPA 8260B	Isopropyl alcohol (2-Propanol, Isopropanol)	SCM	FL	
RCRP	EPA 8260B	Isopropylbenzene	NPW	FL	
RCRP	EPA 8260B	Isopropylbenzene	SCM	FL	
RCRP	EPA 8260B	m+p-xylene	NPW	FL	
RCRP	EPA 8260B	m+p-xylene	SCM	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	Methacrylonitrile	NPW	FL	
RCRP	EPA 8260B	Methacrylonitrile	SCM	FL	
RCRP	EPA 8260B	Methyl bromide (Bromomethane)	SCM	FL	
RCRP	EPA 8260B	Methyl bromide (Bromomethane)	NPW	FL	
RCRP	EPA 8260B	Methyl chloride (Chloromethane)	NPW	FL	
RCRP	EPA 8260B	Methyl chloride (Chloromethane)	SCM	FL	
RCRP	EPA 8260B	Methyl methacrylate	SCM	FL	
RCRP	EPA 8260B	Methyl methacrylate	NPW	FL	
RCRP	EPA 8260B	Methyl tert-butyl ether (MTBE)	SCM	FL	
RCRP	EPA 8260B	Methyl tert-butyl ether (MTBE)	NPW	FL	
RCRP	EPA 8260B	Methylene chloride (Dichloromethane)	SCM	FL	
RCRP	EPA 8260B	Methylene chloride (Dichloromethane)	NPW	FL	
RCRP	EPA 8260B	n-Butyl alcohol (1-Butanol, n-Butanol)	SCM	FL	
RCRP	EPA 8260B	n-Butyl alcohol (1-Butanol, n-Butanol)	NPW	FL	
RCRP	EPA 8260B	n-Butylbenzene	NPW	FL	
RCRP	EPA 8260B	n-Butylbenzene	SCM	FL	
RCRP	EPA 8260B	n-Propanol (1-Propanol)	NPW	FL	
RCRP	EPA 8260B	n-Propanol (1-Propanol)	SCM	FL	
RCRP	EPA 8260B	n-Propylbenzene	NPW	FL	
RCRP	EPA 8260B	n-Propylbenzene	SCM	FL	
RCRP	EPA 8260B	Naphthalene	NPW	FL	
RCRP	EPA 8260B	Naphthalene	SCM	FL	
RCRP	EPA 8260B	o-Xylene	SCM	FL	
RCRP	EPA 8260B	o-Xylene	NPW	FL	
RCRP	EPA 8260B	Propionitrile (Ethyl cyanide)	SCM	FL	
RCRP	EPA 8260B	Propionitrile (Ethyl cyanide)	NPW	FL	
RCRP	EPA 8260B	sec-Butylbenzene	NPW	FL	
RCRP	EPA 8260B	sec-Butylbenzene	SCM	FL	
RCRP	EPA 8260B	Styrene	SCM	FL	
RCRP	EPA 8260B	Styrene	NPW	FL	
RCRP	EPA 8260B	tert-Butyl alcohol	SCM	FL	
RCRP	EPA 8260B	tert-Butyl alcohol	NPW	FL	
RCRP	EPA 8260B	tert-Butylbenzene	NPW	FL	
RCRP	EPA 8260B	tert-Butylbenzene	SCM	FL	
RCRP	EPA 8260B	Tetrachloroethylene (Perchloroethylene)	SCM	FL	
RCRP	EPA 8260B	Tetrachloroethylene (Perchloroethylene)	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	Toluene	SCM	FL	
RCRP	EPA 8260B	Toluene	NPW	FL	
RCRP	EPA 8260B	trans-1,2-Dichloroethylene	SCM	FL	
RCRP	EPA 8260B	trans-1,2-Dichloroethylene	NPW	FL	
RCRP	EPA 8260B	trans-1,3-Dichloropropylene	NPW	FL	
RCRP	EPA 8260B	trans-1,3-Dichloropropylene	SCM	FL	
RCRP	EPA 8260B	trans-1,4-Dichloro-2-butene	SCM	FL	
RCRP	EPA 8260B	trans-1,4-Dichloro-2-butene	NPW	FL	
RCRP	EPA 8260B	Trichloroethene (Trichloroethylene)	SCM	FL	
RCRP	EPA 8260B	Trichloroethene (Trichloroethylene)	NPW	FL	
RCRP	EPA 8260B	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	SCM	FL	
RCRP	EPA 8260B	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	NPW	FL	
RCRP	EPA 8260B	Vinyl acetate	NPW	FL	
RCRP	EPA 8260B	Vinyl acetate	SCM	FL	
RCRP	EPA 8260B	Vinyl chloride	NPW	FL	
RCRP	EPA 8260B	Vinyl chloride	SCM	FL	

EPA 8260C

Preparation Techniques: Purge and trap; Extraction, EPA 1312 SPLP, zero headspace (ZHE); Extraction, EPA 1311 TCLP, zero headspace (ZHE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	NPW	FL	
RCRP	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	SCM	FL	

EPA 8270C

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Extraction, EPA 1312 SPLP, non-volatiles; Extraction, separatory funnel liquid-liquid (LLE); Extraction, ultrasonic; Extraction, pressurized fluid (PFE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	1,2,4,5-Tetrachlorobenzene	NPW	FL	
RCRP	EPA 8270C	1,2,4,5-Tetrachlorobenzene	SCM	FL	
RCRP	EPA 8270C	1,2,4-Trichlorobenzene	SCM	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	1,2,4-Trichlorobenzene	NPW	FL	
RCRP	EPA 8270C	1,2-Dichlorobenzene	NPW	FL	
RCRP	EPA 8270C	1,2-Dichlorobenzene	SCM	FL	
RCRP	EPA 8270C	1,2-Diphenylhydrazine	NPW	FL	
RCRP	EPA 8270C	1,2-Diphenylhydrazine	SCM	FL	
RCRP	EPA 8270C	1,3,5-Trinitrobenzene (1,3,5-TNB)	NPW	FL	
RCRP	EPA 8270C	1,3,5-Trinitrobenzene (1,3,5-TNB)	SCM	FL	
RCRP	EPA 8270C	1,3-Dichlorobenzene	SCM	FL	
RCRP	EPA 8270C	1,3-Dichlorobenzene	NPW	FL	
RCRP	EPA 8270C	1,3-Dinitrobenzene (1,3-DNB)	SCM	FL	
RCRP	EPA 8270C	1,3-Dinitrobenzene (1,3-DNB)	NPW	FL	
RCRP	EPA 8270C	1,4-Dichlorobenzene	SCM	FL	
RCRP	EPA 8270C	1,4-Dichlorobenzene	NPW	FL	
RCRP	EPA 8270C	1,4-Naphthoquinone	NPW	FL	
RCRP	EPA 8270C	1,4-Naphthoquinone	SCM	FL	
RCRP	EPA 8270C	1,4-Phenylenediamine	SCM	FL	
RCRP	EPA 8270C	1,4-Phenylenediamine	NPW	FL	
RCRP	EPA 8270C	1-Naphthylamine	NPW	FL	
RCRP	EPA 8270C	1-Naphthylamine	SCM	FL	
RCRP	EPA 8270C	2,3,4,6-Tetrachlorophenol	SCM	FL	
RCRP	EPA 8270C	2,3,4,6-Tetrachlorophenol	NPW	FL	
RCRP	EPA 8270C	2,4,5-Trichlorophenol	NPW	FL	
RCRP	EPA 8270C	2,4,5-Trichlorophenol	SCM	FL	
RCRP	EPA 8270C	2,4,6-Trichlorophenol	SCM	FL	
RCRP	EPA 8270C	2,4,6-Trichlorophenol	NPW	FL	
RCRP	EPA 8270C	2,4-Dichlorophenol	SCM	FL	
RCRP	EPA 8270C	2,4-Dichlorophenol	NPW	FL	
RCRP	EPA 8270C	2,4-Dimethylphenol	SCM	FL	
RCRP	EPA 8270C	2,4-Dimethylphenol	NPW	FL	
RCRP	EPA 8270C	2,4-Dinitrophenol	SCM	FL	
RCRP	EPA 8270C	2,4-Dinitrophenol	NPW	FL	
RCRP	EPA 8270C	2,4-Dinitrotoluene (2,4-DNT)	SCM	FL	
RCRP	EPA 8270C	2,4-Dinitrotoluene (2,4-DNT)	NPW	FL	
RCRP	EPA 8270C	2,6-Dichlorophenol	NPW	FL	
RCRP	EPA 8270C	2,6-Dichlorophenol	SCM	FL	
RCRP	EPA 8270C	2,6-Dinitrotoluene (2,6-DNT)	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	2,6-Dinitrotoluene (2,6-DNT)	SCM	FL	
RCRP	EPA 8270C	2-Acetylaminofluorene	SCM	FL	
RCRP	EPA 8270C	2-Acetylaminofluorene	NPW	FL	
RCRP	EPA 8270C	2-Chloronaphthalene	NPW	FL	
RCRP	EPA 8270C	2-Chloronaphthalene	SCM	FL	
RCRP	EPA 8270C	2-Chlorophenol	NPW	FL	
RCRP	EPA 8270C	2-Chlorophenol	SCM	FL	
RCRP	EPA 8270C	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	SCM	FL	
RCRP	EPA 8270C	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	NPW	FL	
RCRP	EPA 8270C	2-Methylaniline (o-Toluidine)	SCM	FL	
RCRP	EPA 8270C	2-Methylaniline (o-Toluidine)	NPW	FL	
RCRP	EPA 8270C	2-Methylnaphthalene	SCM	FL	
RCRP	EPA 8270C	2-Methylnaphthalene	NPW	FL	
RCRP	EPA 8270C	2-Methylphenol (o-Cresol)	SCM	FL	
RCRP	EPA 8270C	2-Methylphenol (o-Cresol)	NPW	FL	
RCRP	EPA 8270C	2-Naphthylamine	NPW	FL	
RCRP	EPA 8270C	2-Naphthylamine	SCM	FL	
RCRP	EPA 8270C	2-Nitroaniline	NPW	FL	
RCRP	EPA 8270C	2-Nitroaniline	SCM	FL	
RCRP	EPA 8270C	2-Nitrophenol	SCM	FL	
RCRP	EPA 8270C	2-Nitrophenol	NPW	FL	
RCRP	EPA 8270C	2-Picoline (2-Methylpyridine)	NPW	FL	
RCRP	EPA 8270C	2-Picoline (2-Methylpyridine)	SCM	FL	
RCRP	EPA 8270C	3,3'-Dichlorobenzidine	NPW	FL	
RCRP	EPA 8270C	3,3'-Dichlorobenzidine	SCM	FL	
RCRP	EPA 8270C	3,3'-Dimethylbenzidine	NPW	FL	
RCRP	EPA 8270C	3-Methylcholanthrene	NPW	FL	
RCRP	EPA 8270C	3-Methylcholanthrene	SCM	FL	
RCRP	EPA 8270C	3-Methylphenol (m-Cresol)	SCM	FL	
RCRP	EPA 8270C	3-Methylphenol (m-Cresol)	NPW	FL	
RCRP	EPA 8270C	3-Nitroaniline	SCM	FL	
RCRP	EPA 8270C	3-Nitroaniline	NPW	FL	
RCRP	EPA 8270C	4-Aminobiphenyl	NPW	FL	
RCRP	EPA 8270C	4-Bromophenyl phenyl ether	NPW	FL	
RCRP	EPA 8270C	4-Bromophenyl phenyl ether	SCM	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	4-Chloro-3-methylphenol	SCM	FL	
RCRP	EPA 8270C	4-Chloro-3-methylphenol	NPW	FL	
RCRP	EPA 8270C	4-Chloroaniline	SCM	FL	
RCRP	EPA 8270C	4-Chloroaniline	NPW	FL	
RCRP	EPA 8270C	4-Chlorophenyl phenylether	NPW	FL	
RCRP	EPA 8270C	4-Chlorophenyl phenylether	SCM	FL	
RCRP	EPA 8270C	4-Dimethyl aminoazobenzene	SCM	FL	
RCRP	EPA 8270C	4-Dimethyl aminoazobenzene	NPW	FL	
RCRP	EPA 8270C	4-Methylphenol (p-Cresol)	NPW	FL	
RCRP	EPA 8270C	4-Methylphenol (p-Cresol)	SCM	FL	
RCRP	EPA 8270C	4-Nitroaniline	NPW	FL	
RCRP	EPA 8270C	4-Nitroaniline	SCM	FL	
RCRP	EPA 8270C	4-Nitrophenol	SCM	FL	
RCRP	EPA 8270C	4-Nitrophenol	NPW	FL	
RCRP	EPA 8270C	4-Nitroquinoline 1-oxide	NPW	FL	
RCRP	EPA 8270C	4-Nitroquinoline 1-oxide	SCM	FL	
RCRP	EPA 8270C	5-Nitro-o-toluidine	SCM	FL	
RCRP	EPA 8270C	5-Nitro-o-toluidine	NPW	FL	
RCRP	EPA 8270C	7,12-Dimethylbenz(a) anthracene	SCM	FL	
RCRP	EPA 8270C	7,12-Dimethylbenz(a) anthracene	NPW	FL	
RCRP	EPA 8270C	a-a-Dimethylphenethylamine	NPW	FL	
RCRP	EPA 8270C	a-a-Dimethylphenethylamine	SCM	FL	
RCRP	EPA 8270C	Acenaphthene	SCM	FL	
RCRP	EPA 8270C	Acenaphthene	NPW	FL	
RCRP	EPA 8270C	Acenaphthylene	NPW	FL	
RCRP	EPA 8270C	Acenaphthylene	SCM	FL	
RCRP	EPA 8270C	Acetophenone	NPW	FL	
RCRP	EPA 8270C	Acetophenone	SCM	FL	
RCRP	EPA 8270C	Aniline	NPW	FL	
RCRP	EPA 8270C	Aniline	SCM	FL	
RCRP	EPA 8270C	Anthracene	NPW	FL	
RCRP	EPA 8270C	Anthracene	SCM	FL	
RCRP	EPA 8270C	Aramite	SCM	FL	
RCRP	EPA 8270C	Aramite	NPW	FL	
RCRP	EPA 8270C	Benzidine	NPW	FL	
RCRP	EPA 8270C	Benzidine	SCM	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	Benzo(a)anthracene	NPW	FL	
RCRP	EPA 8270C	Benzo(a)anthracene	SCM	FL	
RCRP	EPA 8270C	Benzo(a)pyrene	NPW	FL	
RCRP	EPA 8270C	Benzo(a)pyrene	SCM	FL	
RCRP	EPA 8270C	Benzo(g,h,i)perylene	SCM	FL	
RCRP	EPA 8270C	Benzo(g,h,i)perylene	NPW	FL	
RCRP	EPA 8270C	Benzo(k)fluoranthene	NPW	FL	
RCRP	EPA 8270C	Benzo(k)fluoranthene	SCM	FL	
RCRP	EPA 8270C	Benzo[b]fluoranthene	NPW	FL	
RCRP	EPA 8270C	Benzo[b]fluoranthene	SCM	FL	
RCRP	EPA 8270C	Benzoic acid	NPW	FL	
RCRP	EPA 8270C	Benzoic acid	SCM	FL	
RCRP	EPA 8270C	Benzyl alcohol	NPW	FL	
RCRP	EPA 8270C	Benzyl alcohol	SCM	FL	
RCRP	EPA 8270C	bis(2-Chloroethoxy)methane	NPW	FL	
RCRP	EPA 8270C	bis(2-Chloroethoxy)methane	SCM	FL	
RCRP	EPA 8270C	bis(2-Chloroethyl) ether	NPW	FL	
RCRP	EPA 8270C	bis(2-Chloroethyl) ether	SCM	FL	
RCRP	EPA 8270C	bis(2-Chloroisopropyl) ether	NPW	FL	
RCRP	EPA 8270C	bis(2-Chloroisopropyl) ether	SCM	FL	
RCRP	EPA 8270C	Butyl benzyl phthalate	SCM	FL	
RCRP	EPA 8270C	Butyl benzyl phthalate	NPW	FL	
RCRP	EPA 8270C	Chlorobenzilate	NPW	FL	
RCRP	EPA 8270C	Chlorobenzilate	SCM	FL	
RCRP	EPA 8270C	Chrysene	SCM	FL	
RCRP	EPA 8270C	Chrysene	NPW	FL	
RCRP	EPA 8270C	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	NPW	FL	
RCRP	EPA 8270C	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	SCM	FL	
RCRP	EPA 8270C	Di-n-butyl phthalate	NPW	FL	
RCRP	EPA 8270C	Di-n-butyl phthalate	SCM	FL	
RCRP	EPA 8270C	Di-n-octyl phthalate	SCM	FL	
RCRP	EPA 8270C	Di-n-octyl phthalate	NPW	FL	
RCRP	EPA 8270C	Diallate	SCM	FL	
RCRP	EPA 8270C	Diallate	NPW	FL	
RCRP	EPA 8270C	Dibenz(a,h) anthracene	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	Dibenz(a,h) anthracene	SCM	FL	
RCRP	EPA 8270C	Dibenzofuran	NPW	FL	
RCRP	EPA 8270C	Dibenzofuran	SCM	FL	
RCRP	EPA 8270C	Diethyl phthalate	SCM	FL	
RCRP	EPA 8270C	Diethyl phthalate	NPW	FL	
RCRP	EPA 8270C	Dimethoate	SCM	FL	
RCRP	EPA 8270C	Dimethoate	NPW	FL	
RCRP	EPA 8270C	Dimethyl phthalate	NPW	FL	
RCRP	EPA 8270C	Dimethyl phthalate	SCM	FL	
RCRP	EPA 8270C	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	SCM	FL	
RCRP	EPA 8270C	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	NPW	FL	
RCRP	EPA 8270C	Disulfoton	NPW	FL	
RCRP	EPA 8270C	Ethyl methanesulfonate	SCM	FL	
RCRP	EPA 8270C	Ethyl methanesulfonate	NPW	FL	
RCRP	EPA 8270C	Famphur	SCM	FL	
RCRP	EPA 8270C	Famphur	NPW	FL	
RCRP	EPA 8270C	Fluoranthene	NPW	FL	
RCRP	EPA 8270C	Fluoranthene	SCM	FL	
RCRP	EPA 8270C	Fluorene	SCM	FL	
RCRP	EPA 8270C	Fluorene	NPW	FL	
RCRP	EPA 8270C	Hexachlorobenzene	NPW	FL	
RCRP	EPA 8270C	Hexachlorobenzene	SCM	FL	
RCRP	EPA 8270C	Hexachlorobutadiene	NPW	FL	
RCRP	EPA 8270C	Hexachlorobutadiene	SCM	FL	
RCRP	EPA 8270C	Hexachlorocyclopentadiene	SCM	FL	
RCRP	EPA 8270C	Hexachlorocyclopentadiene	NPW	FL	
RCRP	EPA 8270C	Hexachloroethane	SCM	FL	
RCRP	EPA 8270C	Hexachloroethane	NPW	FL	
RCRP	EPA 8270C	Hexachloropropene	SCM	FL	
RCRP	EPA 8270C	Hexachloropropene	NPW	FL	
RCRP	EPA 8270C	Indeno(1,2,3-cd) pyrene	NPW	FL	
RCRP	EPA 8270C	Indeno(1,2,3-cd) pyrene	SCM	FL	
RCRP	EPA 8270C	Isodrin	NPW	FL	
RCRP	EPA 8270C	Isodrin	SCM	FL	
RCRP	EPA 8270C	Isophorone	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	Isophorone	SCM	FL	
RCRP	EPA 8270C	Isosafrole	NPW	FL	
RCRP	EPA 8270C	Isosafrole	SCM	FL	
RCRP	EPA 8270C	Kepone	NPW	FL	
RCRP	EPA 8270C	Kepone	SCM	FL	
RCRP	EPA 8270C	Methyl methanesulfonate	NPW	FL	
RCRP	EPA 8270C	Methyl methanesulfonate	SCM	FL	
RCRP	EPA 8270C	Methyl parathion (Parathion, methyl)	SCM	FL	
RCRP	EPA 8270C	Methyl parathion (Parathion, methyl)	NPW	FL	
RCRP	EPA 8270C	n-Nitroso-di-n-butylamine	NPW	FL	
RCRP	EPA 8270C	n-Nitroso-di-n-butylamine	SCM	FL	
RCRP	EPA 8270C	n-Nitrosodi-n-propylamine	NPW	FL	
RCRP	EPA 8270C	n-Nitrosodi-n-propylamine	SCM	FL	
RCRP	EPA 8270C	n-Nitrosodiethylamine	SCM	FL	
RCRP	EPA 8270C	n-Nitrosodiethylamine	NPW	FL	
RCRP	EPA 8270C	n-Nitrosodimethylamine	SCM	FL	
RCRP	EPA 8270C	n-Nitrosodimethylamine	NPW	FL	
RCRP	EPA 8270C	n-Nitrosodiphenylamine	NPW	FL	
RCRP	EPA 8270C	n-Nitrosodiphenylamine	SCM	FL	
RCRP	EPA 8270C	n-Nitrosomorpholine	NPW	FL	
RCRP	EPA 8270C	n-Nitrosomorpholine	SCM	FL	
RCRP	EPA 8270C	n-Nitrosopiperidine	NPW	FL	
RCRP	EPA 8270C	n-Nitrosopiperidine	SCM	FL	
RCRP	EPA 8270C	n-Nitrosopyrrolidine	NPW	FL	
RCRP	EPA 8270C	n-Nitrosopyrrolidine	SCM	FL	
RCRP	EPA 8270C	Naphthalene	SCM	FL	
RCRP	EPA 8270C	Naphthalene	NPW	FL	
RCRP	EPA 8270C	Nitrobenzene	SCM	FL	
RCRP	EPA 8270C	Nitrobenzene	NPW	FL	
RCRP	EPA 8270C	o,o,o-Triethyl phosphorothioate	SCM	FL	
RCRP	EPA 8270C	o,o,o-Triethyl phosphorothioate	NPW	FL	
RCRP	EPA 8270C	Parathion, ethyl	SCM	FL	
RCRP	EPA 8270C	Parathion, ethyl	NPW	FL	
RCRP	EPA 8270C	Pentachlorobenzene	NPW	FL	
RCRP	EPA 8270C	Pentachlorobenzene	SCM	FL	
RCRP	EPA 8270C	Pentachloronitrobenzene	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	Pentachloronitrobenzene	SCM	FL	
RCRP	EPA 8270C	Pentachlorophenol	SCM	FL	
RCRP	EPA 8270C	Pentachlorophenol	NPW	FL	
RCRP	EPA 8270C	Phenacetin	SCM	FL	
RCRP	EPA 8270C	Phenacetin	NPW	FL	
RCRP	EPA 8270C	Phenanthrene	SCM	FL	
RCRP	EPA 8270C	Phenanthrene	NPW	FL	
RCRP	EPA 8270C	Phenol	SCM	FL	
RCRP	EPA 8270C	Phenol	NPW	FL	
RCRP	EPA 8270C	Phorate	NPW	FL	
RCRP	EPA 8270C	Phorate	SCM	FL	
RCRP	EPA 8270C	Pronamide (Kerb)	NPW	FL	
RCRP	EPA 8270C	Pronamide (Kerb)	SCM	FL	
RCRP	EPA 8270C	Pyrene	SCM	FL	
RCRP	EPA 8270C	Pyrene	NPW	FL	
RCRP	EPA 8270C	Pyridine	SCM	FL	
RCRP	EPA 8270C	Pyridine	NPW	FL	
RCRP	EPA 8270C	Safrole	SCM	FL	
RCRP	EPA 8270C	Safrole	NPW	FL	
RCRP	EPA 8270C	Sulfotepp	SCM	FL	
RCRP	EPA 8270C	Sulfotepp	NPW	FL	
RCRP	EPA 8270C	Thionazin (Zinophos)	SCM	FL	
RCRP	EPA 8270C	Thionazin (Zinophos)	NPW	FL	

EPA 8315A

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Extraction, EPA 1312 SPLP, non-volatiles; Extraction, solid phase (SPE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8315A	Acetaldehyde	NPW	FL	
RCRP	EPA 8315A	Crotonaldehyde	NPW	FL	
RCRP	EPA 8315A	Cyclohexanone	NPW	FL	
RCRP	EPA 8315A	Decanal	NPW	FL	
RCRP	EPA 8315A	Formaldehyde	NPW	FL	
RCRP	EPA 8315A	Heptanal	NPW	FL	
RCRP	EPA 8315A	Hexanaldehyde (Hexanal)	NPW	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8315A	m-Tolualdehyde (1,3-Tolualdehyde)	NPW	FL	
RCRP	EPA 8315A	n-Octaldehyde (Octanal)	NPW	FL	
RCRP	EPA 8315A	Nonanal	NPW	FL	
RCRP	EPA 8315A	Propionaldehyde (Propanal)	NPW	FL	
RCRP	EPA 8315A	Valeraldehyde (Pentanal, Pentanaldehyde)	NPW	FL	

EPA 8330

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, ultrasonic;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8330	1,3,5-Trinitrobenzene (1,3,5-TNB)	SCM	FL	
RCRP	EPA 8330	1,3,5-Trinitrobenzene (1,3,5-TNB)	NPW	FL	
RCRP	EPA 8330	1,3-Dinitrobenzene (1,3-DNB)	NPW	FL	
RCRP	EPA 8330	1,3-Dinitrobenzene (1,3-DNB)	SCM	FL	
RCRP	EPA 8330	2,4,6-Trinitrotoluene (2,4,6-TNT)	NPW	FL	
RCRP	EPA 8330	2,4,6-Trinitrotoluene (2,4,6-TNT)	SCM	FL	
RCRP	EPA 8330	2,4-Dinitrotoluene (2,4-DNT)	SCM	FL	
RCRP	EPA 8330	2,4-Dinitrotoluene (2,4-DNT)	NPW	FL	
RCRP	EPA 8330	2,6-Dinitrotoluene (2,6-DNT)	SCM	FL	
RCRP	EPA 8330	2,6-Dinitrotoluene (2,6-DNT)	NPW	FL	
RCRP	EPA 8330	2-Amino-4,6-dinitrotoluene (2-am-dnt)	SCM	FL	
RCRP	EPA 8330	2-Amino-4,6-dinitrotoluene (2-am-dnt)	NPW	FL	
RCRP	EPA 8330	2-Nitrotoluene	NPW	FL	
RCRP	EPA 8330	2-Nitrotoluene	SCM	FL	
RCRP	EPA 8330	3-Nitrotoluene	SCM	FL	
RCRP	EPA 8330	3-Nitrotoluene	NPW	FL	
RCRP	EPA 8330	4-Amino-2,6-dinitrotoluene (4-am-dnt)	NPW	FL	
RCRP	EPA 8330	4-Amino-2,6-dinitrotoluene (4-am-dnt)	SCM	FL	
RCRP	EPA 8330	4-Nitrotoluene	NPW	FL	
RCRP	EPA 8330	4-Nitrotoluene	SCM	FL	
RCRP	EPA 8330	Methyl-2,4,6-trinitrophenylnitramine (tetryl)	NPW	FL	
RCRP	EPA 8330	Methyl-2,4,6-trinitrophenylnitramine (tetryl)	SCM	FL	
RCRP	EPA 8330	Nitrobenzene	NPW	FL	
RCRP	EPA 8330	Nitrobenzene	SCM	FL	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8330	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	NPW	FL	
RCRP	EPA 8330	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	SCM	FL	
RCRP	EPA 8330	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	NPW	FL	
RCRP	EPA 8330	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	SCM	FL	

EPA 9013A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9013A	Cyanide	SCM	FL	

EPA 9020B

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9020B	Total Organic Halides (TOX)	NPW	FL	

EPA 9034

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9034	Sulfide	SCM	FL	
RCRP	EPA 9034	Sulfide	NPW	FL	

EPA 9038

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9038	Sulfate	NPW	FL	

EPA 9040C

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9040C	pH	NPW	FL	

EPA 9045D

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9045D	pH	SCM	FL	

EPA 9056A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9056A	Chloride	NPW	FL	
RCRP	EPA 9056A	Chloride	SCM	FL	
RCRP	EPA 9056A	Fluoride	SCM	FL	
RCRP	EPA 9056A	Fluoride	NPW	FL	
RCRP	EPA 9056A	Nitrate	NPW	FL	
RCRP	EPA 9056A	Nitrate	SCM	FL	
RCRP	EPA 9056A	Nitrite	NPW	FL	
RCRP	EPA 9056A	Nitrite	SCM	FL	
RCRP	EPA 9056A	Sulfate	SCM	FL	
RCRP	EPA 9056A	Sulfate	NPW	FL	

EPA 9065

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9065	Total Phenolics	NPW	FL	
RCRP	EPA 9065	Total Phenolics	SCM	FL	

EPA 9070A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9070A	Oil & Grease	NPW	FL	

EPA 9071B

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9071B	n-Hexane Extractable Material (O&G)	SCM	FL	
RCRP	EPA 9071B	Oil & Grease	SCM	FL	

EPA 9095B

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9095B	Paint Filter Liquids Test	SCM	FL	

EPA RSK-175 (GC/FID)

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA RSK-175 (GC/FID)	Ethane	NPW	FL	
RCRP	EPA RSK-175 (GC/FID)	Ethene	NPW	FL	
RCRP	EPA RSK-175 (GC/FID)	Methane	NPW	FL	

Walkley-Black

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	Walkley-Black	Total Organic Carbon	SCM	FL	

Underground Storage Tank Program

WI(95) DRO

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	WI(95) DRO	Diesel range organics (DRO)	NPW	FL	
USTP	WI(95) DRO	Diesel range organics (DRO)	SCM	FL	

WI(95) GRO

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	WI(95) GRO	Gasoline range organics (GRO)	SCM	FL	
USTP	WI(95) GRO	Gasoline range organics (GRO)	NPW	FL	

WI(95) GRO

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	WI(95) GRO	Petroleum Volatile Organic Compounds (PVOC)	SCM	FL	
USTP	WI(95) GRO	Petroleum Volatile Organic Compounds (PVOC)	NPW	FL	

Note: Method beginning with "SM" refer to the approved editions of Standard methods for the Examination of Water and Wastes. Approved methods are listed in the applicable parts of Title 40 of the Code of Federal Regulations (including its subsequent Federal Register updates), MN Statutes and Rules, and state-issued permits.

**APPENDIX C
LABORATORY CONTROL LIMITS**

Analyte	CAS No.	Matrix	LCS, MS, and MSD Recovery and RPD Limits			Source For QC Limits
			LCL	UCL	RPD	
			%	%	%	
Matrix: Groundwater						
VOCs:						
1,1,1-Trichloroethane	71-55-6	Groundwater	65	130	30	DoD QSM version 4.2
1,1-Dichloroethane	75-34-3	Groundwater	70	135	30	DoD QSM version 4.2
1,1-Dichloroethene	75-35-4	Groundwater	70	130	30	DoD QSM version 4.2
cis-1,2-Dichloroethene	156-59-2	Groundwater	70	125	30	DoD QSM version 4.2
Tetrachloroethene	127-18-4	Groundwater	45	150	30	DoD QSM version 4.2
trans-1,2-Dichloroethene	156-60-5	Groundwater	60	140	30	DoD QSM version 4.2
Trichloroethene	79-01-6	Groundwater	70	125	30	DoD QSM version 4.2
Vinyl chloride	75-01-4	Groundwater	50	145	30	DoD QSM version 4.2
VOC Surrogates:						
Dibromofluoromethane	1868-53-7	Groundwater	85	115	N/A	DoD QSM version 4.2
1,2-Dichloroethane-d4	17060-07-0	Groundwater	70	120	N/A	DoD QSM version 4.2
Toluene-d8	2037-26-5	Groundwater	85	120	N/A	DoD QSM version 4.2
4-Bromofluorobenzene	460-00-4	Groundwater	75	120	N/A	DoD QSM version 4.2
Non-VOC Analytes:						
Sulfate	18785-72-3	Groundwater	80	120	15	DoD QSM version 4.2
Nitrate	14797-55-8	Groundwater	80	120	15	DoD QSM version 4.2
Total Iron	7439-89-6	Groundwater	80	120	20	DoD QSM version 4.2
Dissolved Iron	7439-89-6	Groundwater	80	120	20	DoD QSM version 4.2
Methane	74-82-8	Groundwater	70	116	20	Laboratory Limits
Ethane	74-84-0	Groundwater	67	122	20	Laboratory Limits
Ethene	74-85-1	Groundwater	67	121	20	Laboratory Limits
Matrix: Soil						
VOCs:						
1,1,1-Trichloroethane	71-55-6	Soil	70	135	30	DoD QSM version 4.2
1,1-Dichloroethane	75-34-3	Soil	75	125	30	DoD QSM version 4.2
1,1-Dichloroethene	75-35-4	Soil	65	135	30	DoD QSM version 4.2
cis-1,2-Dichloroethene	154-59-2	Soil	65	125	30	DoD QSM version 4.2
Tetrachloroethene	127-18-4	Soil	65	140	30	DoD QSM version 4.2
trans-1,2-Dichloroethene	156-60-5	Soil	65	135	30	DoD QSM version 4.2
Trichloroethene	79-01-6	Soil	75	125	30	DoD QSM version 4.2
Vinyl chloride	75-01-4	Soil	60	125	30	DoD QSM version 4.2
VOC Surrogates:						
4-Bromofluorobenzene		Soil	85	120		DoD QSM version 4.2
Toluene-d8		Soil	85	115		DoD QSM version 4.2
Non-VOC Analytes:						
Iron	7439-89-6	Soil	80	120	20	DoD QSM version 4.2
Total organic carbon	7440-44-0	Soil	85	115	20	Laboratory Limits

**APPENDIX C
LABORATORY CONTROL LIMITS**

Analyte	CAS No.	Matrix	LCS, MS, and MSD Recovery and RPD Limits			Source For QC Limits
			LCL	UCL	RPD	
			%	%	%	
Matrix: IDW – Soil and Water						
TCLP Volatile Organic Compounds						
1,1-Dichloroethene	75-35-4	TCLP Liquid	71	129	20	Laboratory Limits
1,2-Dichloroethane	107-06-2	TCLP Liquid	78	125	20	Laboratory Limits
2-Butanone (MEK)	78-93-3	TCLP Liquid	32	178	20	Laboratory Limits
Benzene	71-43-2	TCLP Liquid	77	122	20	Laboratory Limits
Carbon tetrachloride	56-23-5	TCLP Liquid	77	132	20	Laboratory Limits
Chlorobenzene	108-90-7	TCLP Liquid	76	128	20	Laboratory Limits
Chloroform	67-66-3	TCLP Liquid	78	127	20	Laboratory Limits
Tetrachloroethene	127-18-4	TCLP Liquid	78	131	20	Laboratory Limits
Trichloroethene	79-01-6	TCLP Liquid	72	129	20	Laboratory Limits
Vinyl chloride	75-01-4	TCLP Liquid	66	139	20	Laboratory Limits
VOC Surrogates						
Dibromofluoromethane	1868-53-7	TCLP Liquid	79	124	N/A	Laboratory Limits
1,2-Dichloroethane-d4	17060-07-0	TCLP Liquid	75	128	N/A	Laboratory Limits
Toluene-d8	2037-26-5	TCLP Liquid	87	113	N/A	Laboratory Limits
4-Bromofluorobenzene	460-00-4	TCLP Liquid	70	121	N/A	Laboratory Limits
TCLP Semivolatile Organic Compounds						
1,4-Dichlorobenzene	106-46-7	TCLP Liquid	36	120	20	Laboratory Limits
2,4,5-Trichlorophenol	95-95-4	TCLP Liquid	41	135	20	Laboratory Limits
2,4,6-Trichlorophenol	88-06-2	TCLP Liquid	36	137	20	Laboratory Limits
2,4-DNT	121-14-2	TCLP Liquid	42	125	20	Laboratory Limits
2-Methylphenol	95-48-7	TCLP Liquid	37	125	20	Laboratory Limits
4-Methylphenol	8001-28-3	TCLP Liquid	45	125	20	Laboratory Limits
Hexachlorobenzene	118-74-1	TCLP Liquid	26	141	20	Laboratory Limits
Hexachlorobutadiene	87-68-3	TCLP Liquid	39	120	20	Laboratory Limits
Hexachloroethane	67-72-1	TCLP Liquid	34	125	20	Laboratory Limits
Nitrobenzene	98-95-3	TCLP Liquid	52	129	20	Laboratory Limits
Pentachlorophenol	87-86-5	TCLP Liquid	39	112	20	Laboratory Limits
Pyridine	110-86-1	TCLP Liquid	5	120	20	Laboratory Limits
TCLP SVOC Surrogates						
2-Fluorophenol	367-12-4	TCLP Liquid	20	121	N/A	Laboratory Limits
Phenol-d6	13127-88-3	TCLP Liquid	10	105	N/A	Laboratory Limits
Nitrobenzene-d5	4165-60-0	TCLP Liquid	38	141	N/A	Laboratory Limits
2-Fluorobiphenyl	321-60-8	TCLP Liquid	41	132	N/A	Laboratory Limits
2,4,6-Tribromophenol	118-79-6	TCLP Liquid	20	142	N/A	Laboratory Limits
o-Terphenyl	84-15-1	TCLP Liquid	39	148	N/A	Laboratory Limits
TCLP Metals						
Arsenic	7440-38-2	TCLP Liquid	80	120	20	DoD QSM version 4.2
Barium	7440-39-3	TCLP Liquid	80	120	20	DoD QSM version 4.2
Cadmium	7440-43-9	TCLP Liquid	80	120	20	DoD QSM version 4.2
Chromium	7440-47-3	TCLP Liquid	80	120	20	DoD QSM version 4.2
Lead	7439-92-1	TCLP Liquid	80	120	20	DoD QSM version 4.2
Mercury	7439-97-6	TCLP Liquid	80	120	20	DoD QSM version 4.2
Selenium	7782-49-2	TCLP Liquid	80	120	20	DoD QSM version 4.2
Silver	7440-22-4	TCLP Liquid	80	120	20	DoD QSM version 4.2

**APPENDIX C
LABORATORY CONTROL LIMITS**

Analyte	CAS No.	Matrix	LCS, MS, and MSD Recovery and RPD Limits			Source For QC Limits
			LCL	UCL	RPD	
			%	%	%	
TCLP Pesticides:						
Chlordane (Technical)	12789-03-6	TCLP Liquid	11	175	20	Laboratory Limits
Endrin	72-20-8	TCLP Liquid	64	129	20	Laboratory Limits
Heptachlor	76-44-8	TCLP Liquid	61	116	20	Laboratory Limits
Heptachlor epoxide	1024-57-3	TCLP Liquid	68	117	20	Laboratory Limits
Lindane (gamma-BHC)	58-89-9	TCLP Liquid	67	120	20	Laboratory Limits
Methoxychlor	72-43-5	TCLP Liquid	71	131	20	Laboratory Limits
Toxaphene	8001-35-2	TCLP Liquid	59	145	20	Laboratory Limits
TCLP Pesticide Surrogates						
Tetrachloro-m-xylene	877-09-8	TCLP Liquid	29	137	N/A	Laboratory Limits
Decachlorobiphenyl	2051-24-3	TCLP Liquid	30	134	N/A	Laboratory Limits
Others:						
PCBs (Soil)	1336-36-3	Soil IDW				
PCB-1016	12674-11-2	Soil IDW	40	140	30	DoD QSM version 4.2
PCB-1221	11104-28-2	Soil IDW	N/A	N/A	N/A	
PCB-1232	11141-16-5	Soil IDW	N/A	N/A	N/A	
PCB-1242	53469-21-9	Soil IDW	N/A	N/A	N/A	
PCB-1248	12672-29-6	Soil IDW	N/A	N/A	N/A	
PCB-1254	11097-69-1	Soil IDW	N/A	N/A	N/A	
PCB-1260	11096-82-5	Soil IDW	60	130	30	DoD QSM version 4.2
Surrogates						
Tetrachloro-m-xylene	877-09-8	Soil IDW	32	129	N/A	DoD QSM version 4.2
Decachlorobiphenyl	2051-24-3	Soil IDW	60	125	N/A	Laboratory Limits
PCBs (Water)						
PCBs (Water)	1336-36-3	Water IDW				
PCB-1016	12674-11-2	Water IDW	25	145	30	DoD QSM version 4.2
PCB-1221	11104-28-2	Water IDW	N/A	N/A	N/A	
PCB-1232	11141-16-5	Water IDW	N/A	N/A	N/A	
PCB-1242	53469-21-9	Water IDW	N/A	N/A	N/A	
PCB-1248	12672-29-6	Water IDW	N/A	N/A	N/A	
PCB-1254	11097-69-1	Water IDW	N/A	N/A	N/A	
PCB-1260	11096-82-5	Water IDW	30	145	30	DoD QSM version 4.2
Surrogates						
Tetrachloro-m-xylene	877-09-8	Water IDW	36	114	N/A	Laboratory Limits
Decachlorobiphenyl	2051-24-3	Water IDW	40	135	N/A	DoD QSM version 4.2
pH						
pH (Soil)	N/A	Soil IDW	N/A	N/A	20	Laboratory Limits
pH Water)	N/A	Water IDW	N/A	N/A	20	Laboratory Limits
Paint Filter Test	N/A	Soil IDW	N/A	N/A	20	Laboratory Limits
Ignitability						
Ignitability (Soil)	N/A	Soil IDW	N/A	N/A	20	Laboratory Limits
Flashpoint (Water)	N/A	Water IDW	N/A	N/A	20	Laboratory Limits



STANDARD OPERATING PROCEDURE

Inductively Coupled Plasma Atomic Emission Spectroscopy Perkin Elmer Optima-3300DV/5300DV

SW-846 Method 6010C

APPROVALS:

Area Supervisor: _____

Denise S. Coffey
Denise S. Coffey

Date: 9-12-11

QA Officer: _____

Tam C. B...
Tam C. B...

Date: 9-12-11

President: _____

Douglas E. Krasunas
Douglas E. Krasunas

Date: 9-12-2011

Procedure Number: GR-01-100

Revision Number: 5.9

Date Initiated: 4/4/06

Effective Date: 6/20/11

Date Revised: 7/22/11

Pages Revised: All

By: Kari L. Visser

Total Number of Pages: 52

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If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
<u>10-04-12</u>	<i>Denise Coffey</i>	<u>10-04-13</u>
_____	_____	_____
_____	_____	_____

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 IV, Method 6010C, "Inductively Coupled Plasma-Atomic Emission Spectrometry", Revision 3, February 2007*

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 polychromator, 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.

4.0 PARAMETER OR COMPOUND LIST

4.1 This procedure may be used for the analysis of the following metals:

Aluminum	Molybdenum
Arsenic	Nickel
Barium	Phosphorus
Beryllium	Potassium
Boron	Selenium
Cadmium	Silicon
Calcium	Silver
Cerium**	Sodium
Chromium	Strontium
Cobalt	Sulfur *
Copper	Thallium
Iron	Tin
Lead	Titanium
Lithium	Vanadium
Magnesium	Zinc
Manganese	

* Optima 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-01-131, *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
- 5.8 *ICP Winlab Software Guide*, Revision E, June 1997
- 5.9 *Optima 3000 Family Hardware Guide*, Release B, August 1997

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 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 3300) shows the interferences that have been documented for the Optima 3300. 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 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, Chromium, Iron, Manganese, and Vanadium interfere with Magnesium. Iron is in IFB1 and the rest are in IFB2, so Magnesium is in IFB2
- 6.1.4.3 To further validate interelement correction factors, all elements are monitored in each IFA solution to confirm that no false positive or negative greater than 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. The Optima 3300DV, and 5300DV

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 the Optima 3300DV and 5300DV 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 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 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 analysis by ICP. Gloves, laboratory coats and approved safety glasses must be worn at all times when handling samples and/or chemicals. Refer to the Material Safety Data Sheets (MSDS) for information on these or any other chemical utilized in this procedure. All MSDS documents are located on the laboratory intranet library in pdf form.

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 0–6° 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. 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 polychromator 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. 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 AEE 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 between 15 and 20°C and pressure must be between 45 and 80 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.

Note: Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument

- 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. These tubes must stand upright within the autosampler rack without tipping or leaning.
- 9.6 Autopipettors capable of delivering 50-1000 μ L.
- 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 filters on the Optimas that must be cleaned regularly.
- 10.2.1 Remove the filters from the holders. Some filters have covers that pop off. These filters can be washed. Some filters slide up and out of the holder. These filters need to be purchased and replaced.
- 10.2.2 To wash the filters, use 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 10.5 for instructions on assembling and disassembling 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 Laboratory Reagent 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 Laboratory Reagent Water from the MilliQ system.

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. N₂ 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 concentrated hydrochloric acid to one part concentrated nitric acid. This is used to clean probes 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 12.6.

- 12.5.1 Rinse four clean 500 mL class A volumetric flasks with laboratory reagent water a minimum of three times.
- 12.5.2 Place 100 mL Laboratory reagent water into each flask.
- 12.5.3 Pipette the correct amounts of acid into the flasks depending on the final acid concentration desired.
- 12.5.3.1 For 10% HCl/5% HNO₃, place 50 mL HCl into the flask and swirl. When the mixing lines are gone from solution, add 25 mL HNO₃ to the flask and swirl until the mixing lines are gone. This is for solid samples.
- 12.5.3.2 For 5% HCl/5% HNO₃, place 25 mL HCl into each flask and swirl. When the mixing lines are gone from solution, add 25 mL HNO₃ 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% HNO₃, place 2.5 mL HNO₃ 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, 50 µL of 10000 mg/L Ce 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, 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, 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 Laboratory reagent 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 Laboratory reagent water a minimum of three times.
- 12.6.2 Place 25 mL Laboratory reagent water into each flask.

- 12.6.3 Pipette the correct amounts of acid into the flasks depending on the final acid concentration desired.
- 12.6.4 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 Laboratory reagent 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 3.6, 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 IFA-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 Laboratory reagent 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 1.0 mL each of 10000 mg/L Cd, Ni, Pb, Zn
- 12.7.2.3.2 0.5 mL each of 10000 mg/L Ba, Co, Cr, Cu, Mn, V
- 12.7.2.3.3 10 mL of 1000 mg/L Ag
- 12.7.2.3.4 0.05 mL of 10000 mg/L Be

- 12.7.2.4 Dilute each flask to the volumetric mark with Laboratory reagent 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 Laboratory reagent water a minimum of three times. Place 2 mL HNO₃ 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.
- 12.7.3.2.1 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 Laboratory reagent 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 Laboratory reagent water a minimum of three times. Place 2 mL HNO₃ 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 1.0 mL of 10,000 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 the flask to the volumetric mark with Laboratory reagent water and mix well. Do not transfer to a bottle. 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 a clean bottle and label to describe the 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 Laboratory reagent water a minimum of three times.

12.8.2 Place about 100 mL Laboratory reagent water into the flask.

12.8.3 Pipette the correct amounts of acid into the flask depending on the final acid concentration desired.

12.8.4 Pipette 10 mL of the solution Interf-1 into the flask and swirl.

12.8.5 Dilute the flask to the volumetric mark with Laboratory reagent water and mix well. Do not transfer to a bottle. Adding the water generates heat and a final volume adjustment must be made after the flask returns 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 a clean bottle and label to describe the bottle's contents.

12.8.8 Refer to attachment 23.4 (IFA Working Solution)

12.9 Prepare the interelement check standard solution IFA-2.

12.9.1 Rinse a clean 500 mL Class A volumetric flask with Laboratory reagent water a minimum of three times.

12.9.2 Place 100 mL laboratory reagent water into the flask.

12.9.3 Pipette the correct amount of acid into the flask depending on the final acid concentration desired.

12.9.4 Pipette 10 mL of the solution IFA2 – Interferent into the flask.

12.9.5 Dilute the flask to the volumetric mark with Laboratory reagent water and mix well. Do not transfer to a bottle. Adding the water generates heat and a final volume adjustment must be made after the flask returns 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.

12.9.10 Refer to Attachment 23.5 (IFA-2 Working Solution)

12.10 Prepare the interelement check standard solution IFB-1.

- 12.10.1 Rinse a clean 500 mL Class A volumetric flask with Laboratory reagent water a minimum of three times.
- 12.10.2 Place 100 mL Laboratory reagent water into the flask.
- 12.10.3 Pipette the correct amount of acid into the flask depending on the final acid concentration desired.
- 12.10.4 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 the flask to the volumetric mark with Laboratory reagent water and mix well. Do not transfer to a bottle. Adding the water generates heat and a final volume adjustment must be made after the flask returns 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 a clean bottle 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 Laboratory reagent water a minimum of three times.
- 12.11.2 Place 100 mL Laboratory reagent water into a flask.
- 12.11.3 Pipette the correct amount of acid into the flask depending on the final acid concentration desired.
- 12.11.4 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 the flask to the volumetric mark with Laboratory reagent water and mix well. Do not transfer to a bottle. Adding the water generates heat and a final volume adjustment must be made after the flask returns 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 a clean bottles and label to describe the 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 Laboratory reagent water a minimum of three times.
- 12.12.2 Place about 100 mL Laboratory reagent water into the flask.
- 12.12.3 Pipette the correct amounts of acid into the flask depending on the final acid concentration desired.
- 12.12.4 Pipette 0.5 mL of the CRL 1 Stock and 0.5 mL of the CRL 2 Stock into the flask.
- 12.12.6 Dilute the flask to the volumetric mark with Laboratory reagent water and mix well. Do not transfer to a bottle. Adding the water generates heat and a final volume adjustment must be made after the flask returns to room temperature.
- 12.12.7 Update the standard numbers in element.
- 12.12.8 After the final volume adjustment, pour the flask contents into a clean bottle and label to describe the bottle's contents.
- 12.12.9 Refer to Attachment 23.13
- 12.13 Prepare the internal standard solution.
- 12.13.1 Rinse a clean 1000 mL Class A volumetric flask with Laboratory reagent water a minimum of three times.
- 12.13.2 Place 100 mL Laboratory reagent water into the flask.
- 12.13.3 Pipette the correct amounts of acid into the flask depending on the final acid concentration desired.
- 12.13.4 Use the volumes listed to prepare concentrations for the instrument being used:
- 12.13.4.1 Pipette 0.5 mL of 10000 mg/L Sc stock standard and 1.0 mL of 10000 mg/L Y stock standard into the flask for the Optima 3300DV.
- 12.13.4.2 Pipette 3.0 mL of 10000 mg/L Sc stock standard and 0.30 mL of 10000 mg/L Y stock standard into the flask for the Optima 5300DV.
- 12.13.5 Dilute each flask to the volumetric mark with Laboratory reagent 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, 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 Sn, Si

12.14.1.2.2 100 mg/L each of Ag, Mo, Ti

12.14.2 The working SCV is prepared fresh daily.

12.14.2.1 Pipet 50 μ L of each SCV stock into 9.9 mL of 10% HCl/5% HNO₃ reagent blank. This is for solid samples.

12.14.2.2 Pipet 50 μ L of each SCV stock into 9.9 mL of 5% HCl/5% HNO₃ reagent blank. This is for digested aqueous samples.

12.14.2.3 Pipet 50 μ L of each SCV stock into 9.9 mL of 0.5% HNO₃ reagent blank. This is for non-digested aqueous samples.

12.14.3 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 TCLP and SPLP Leachates 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 3300DV and 5300DV must be calibrated for every analytical run or every 24 hours, whichever is more frequent.

- 14.1.1 At least once per week or after instrument maintenance, 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". For dual view instruments, both radial and axial views must be optimized.
- 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 Attachment 23.6 (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, initial QC followed by samples and continuing QC is performed.
- 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 continuing calibration verification (CCB). The CCB is a reagent blank having the same acid concentration as the standards. The absolute value of the mean CCB 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 (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) standard 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. 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, 3300DV and 5300DV 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 the 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

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Perkin Elmer OPTIMA-3000/3300DV/5300DV
SW-846 Method 6010C

Revision Number: 5.9

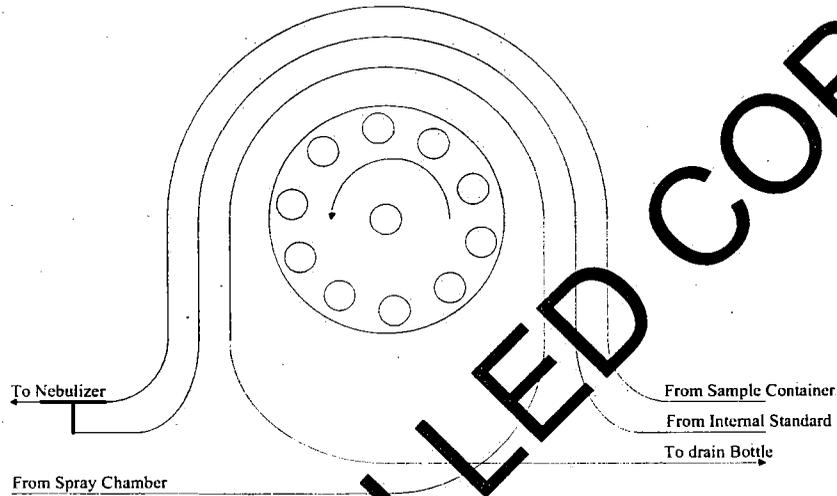
SOP Number: GR-01-100

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Date Revised: 7/22/11

Date Initiated: 4/4/96

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 Refer to Attachment 23.9, Table 8, Element Conditions for the Optima 3000. Dashes indicate no background subtraction point used for this interval.
- 15.8 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, 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.
- 15.9 Refer to Attachment 23.10, Table 9, ID/WT Sheet Setup for Instrument Calibration and Verification
- 15.10 Set up the analysis.
- 15.10.1 Enter the analysis sequence.
 - 15.10.2 Analyze samples.
 - 15.10.3 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.

$$C_{\text{Final}} = C_{\text{Instr}} * DF_{\text{Digest}} * DF_{\text{Subseq}}$$

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:

17.1.1 Instrument 116 and 311 (Optima 3300DV, 5300DV)

17.1.1.1 Click on "File", "Utilities", then "Data Manager".

17.1.1.2 Highlight the dataset to be exported then click "Export".

- 17.1.1.3 Click "New Design", then "Next" and then "Next" again on the two following windows without making any changes.
- 17.1.1.4 Change the dataset file name extension to ".prn".
- 17.1.1.5 Change the directory to groups/LabMe/116(311)/current month/export then click "next".
- 17.1.1.6 Highlight all available parameters and click "Add" then "Next". Repeat for the following page.
- 17.1.1.7 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 initially, after interelement correction factors are updated and after significant changes to the instrument. Calculate the IDL as follows:
- 18.3.1 Analyze seven sequential aliquots of matrix-matched 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.
- 18.4.3 Refer to Addendum 23.15 Generating Inter-Element Correction Factors (IEC) for ICP
- 18.5 Dilution is not required for samples/digestates exceeding the calibration range until 90% of instrument linearity is exceeded (Section 18.3).
- 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 ICV/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 23.1, Table 10, Spiking Solutions Final Concentrations

18.8.3 Calculate the percent recovery of the MS and MSD as follows:

$$\%R = \frac{C_{\text{Spike}} - C_{\text{Orig}}}{\text{Spike Quantity}} \times 100$$

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,

$$\%RPD = \frac{C_{\text{MSD}} - C_{\text{MS}}}{(C_{\text{MSD}} + C_{\text{MS}})/2} \times 100$$

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 MS as read from the raw data printout

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 $\pm 10\%$ of the original determination. If not, then a chemical or physical interference effect should be suspected.

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 $\leq 20\%$ RPD.

18.10 Method of Standard Additions (MSA)

18.10.1 The MSA technique must be performed on all TCEP 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 in accordance with the procedure. Using the regression program located in the spreadsheets folder of the laboratory intranet library, plot absorbance vs 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 23.14.

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 ≥ 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 90 to 125% 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 with second-source standards 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 $\leq 20\%$.
- 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 used 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 demonstration of capability spreadsheets to the quality assurance department for data and training documentation.
- 19.2 A Method Detection Limit (MDL) study must be performed annually on every instrument using this procedure and for each digestion procedure 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.
- 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 IV, Method 6010C, "Inductively Coupled Plasma-Atomic Emission Spectrometry", Revision 3, February 2007*

23.0 ATTACHMENTS

- 23.1 Table 1, Nominal Reporting Limits for the Optima 3000
- 23.2 Table 1A, Analytical Wavelengths and Nominal Reporting Limits for the Optima 3000
- 23.3 Table 2, ICP-AES Inter-element Correction Factors for the Optima 3000
- 23.4 Table 3, IFA1 Working Solution
- 23.5 Table 4, IFA2 Working Solution
- 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, Run Sequence 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, CRL CRL Working Standard Concentrations
- 23.14 Table 13, Method of Standard Additions Curve Example
- 23.15 Generating Inter-Element Correction Factors (IEC) For ICP (Addendum)

Attachment 23.1
 Table 1
 Nominal Reporting Limits for the Optima 3000

Element	Reporting Limits (ug/L)	Reporting Limits (mg/Kg)
Aluminum	50.0	10.0
Arsenic	100.0	10.0
Barium	10.0	1.0
Beryllium	1.0	1.0
Boron	100.0	10.0
Cadmium	10.0	1.0
Calcium	500.0	50.0
Chromium	50.0	5.0
Cobalt	10.0	10.0
Copper	10.0	1.0
Iron	10.0	1.0
Lead	50.0	5.0
Lithium	3.0	1.0
Magnesium	500.0	50.0
Manganese	10.0	1.0
Molybdenum	100.0	10.0
Nickel	10.0	1.0
Potassium	100.0	50.0
Selenium	100.0	10.0
Silicon	500.0	50.0
Silver	10.0	1.0
Sodium	500.0	50.0
Srondium	50.0	5.0
Sulfur	100.0	10.0
Thallium	100.0	10.0
Tin	200.0	20.0
Titanium	100.0	10.0
Vanadium	10.0	1.0
Zinc	20.0	1.0

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Attachment 23.2
Table 1A
Analytical Wavelengths and Nominal Reporting Limits for the Optima 5300

Element	Wavelength (nm)	View	Reporting Limits (ug/L)	Reporting Limits (mg/g)
Aluminum	396.154	Radial	50.0	10.0
Arsenic	198.979	Axial	100.0	10.0
Barium	455.401	Radial	10.0	1.0
Beryllium	234.861	Axial	1.0	1.0
Boron	249.577	Axial	100.0	10.0
Cadmium	214.435	Axial	10.0	1.0
Calcium	315.887	Radial	50.0	50.0
Chromium	268.557	Axial	50.0	5.0
Cobalt	238.513	Axial	10.0	10.0
Copper	327.397	Axial	10.0	1.0
Iron	238.201	Radial	10.0	1.0
Lead	238.351	Axial	50.0	5.0
Lithium	570.782	Radial	5.0	1.0
Magnesium	279.074	Radial	500.0	50.0
Manganese	257.606	Axial	10.0	1.0
Molybdenum	282.93	Radial	100.0	10.0
Nickel	231.503	Axial	10.0	1.0
Potassium	766.494	Radial	100.0	50.0
Scandium	867.763	Radial	NA	NA
Selenium	138.027	Axial	100.0	10.0
Silicon	251.505	Axial	500.0	50.0
Silver	313.067	Axial	10.0	1.0
Sodium	588.998	Radial	500.0	50.0
Strontium	407.769	Radial	50.0	5.0
Tantalum	181.371	Axial	100.0	10.0
Thallium	190.792	Axial	100.0	10.0
Tin	189.927	Axial	200.0	20.0
Titanium	334.339	Radial	100.0	10.0
Vanadium	292.399	Axial	10.0	1.0
Yttrium	360.071	Axial	NA	NA
Zinc	206.198	Axial	20.0	1.0

Attachment 23.3

Table 2

ICP-AES Interelement Correction Factors for the Optima 3300

Analyte	Wavelength (nm)	Interelement Correction Factors For:										
		Al	Ca	Co	Cr	Cu	Fe	Mg	Mn	Mo	Ni	
Aluminum	396.146	N/A	0.36189	-0.46723	-0.309539						29.8125	
Antimony	217.584		-0.42135	-2.7740	18.1183						-24.8072	
Arsenic	188.979		-1.03742		2.20856				0.640527		3.72529	
Barium	455.405			-0.2425								
Beryllium	234.863				0.004498	0.00231335	0.0989343					0.0092764
Boron	249.769						1.46902					
Cadmium	214.436	0.0517453	0.0154087	N/A			0.0620319				-0.0616759	0.21251
Calcium	315.874	0.163209	N/A	0.729633	2.00001	0.98688	0.22829	0.151176	0.143829	0.862722	-4.08617	0.148212
Chromium	205.563		0.0258892	-0.127877		N/A			0.0675095		0.542364	0.720741
Cobalt	228.616	-0.0356691		-0.136622	-0.0795149	N/A	0.249906	-0.0253156	0.108988			-0.156742
Copper	324.741		-0.115653	0.400741		0.62284	-0.03487	N/A		-0.0268978	0.770813	0.105789
Iron	238.204				0.0297433	-56.0573	0.230777		N/A			
Lead	220.353	-0.150982			1.55371	0.73085		1.1000	0.114716			-3.05316
Lithium	670.784											
Magnesium	279.071	0.0190964	0.00520197	0.0787193	-0.227277	-2.00881	-0.249691	0.0207466	-0.326616	N/A	-0.790727	0.0857341
Manganese	257.610				0.0975289						N/A	
Molybdenum	202.031								0.264224		N/A	
Nickel	231.604				0.111248	1.73406			-0.020488		-1.96666	N/A
Phosphorus	178.226											
Potassium	766.490											
Selenium	196.026		-0.361052		-2.60609							
Silver	328.068				-3.83659			0.0383737	-0.01630	-0.0120237	0.137698	-0.111102
Silicon	251.611											7.64627
Sodium	589.592											
Strontium	407.771				-0.572852							
Thallium	190.801		-0.62019		1.31816	8.27783	0.43259		-0.241055		0.160093	-0.211632
Tin	189.927				0.273546							
Titanium	334.946						0.288862					
Vanadium	292.395				0.160497		0.290026		0.117451		-0.156177	-10.0212
Zinc	206.200				-0.0270792		1.65167					

Attachment 23.3
 Table 2 Continued

ICP-AES Interelement Correction Factors for the Optima 3300

Analyte	Wavelength (nm)	Interelement Correction Factors For:																		
		Ti	V	Cr	Mn	Fe	Ni	Cu	Zn	Pb	Ag									
Aluminum	396.146	0.994173																		
Antimony	317.584	1.53504																		
Arsenic	188.979																			
Barium	455.403	-0.309621																		
Beryllium	234.863																			
Boron	249.769																			
Cadmium	214.436		-0.107934																	
Calcium	313.874	0.833329	0.0890178																	
Chromium	305.563	0.102749	0.0879189																	
Cobalt	228.616	1.68283																		
Copper	324.741	0.346694	-0.309574																	
Iron	238.304		0.278988																	
Lead	220.353																			
Lithium	670.784																			
Magnesium	279.071	0.244454	0.257188																	
Manganese	257.610		-0.0463747																	
Molybdenum	202.031		-0.423097																	
Nickel	231.604	0.12548																		
Phosphorus	178.226																			
Potassium	766.490																			
Selenium	196.026																			
Silver	328.068		-0.968323																	
Silicon	251.611	73.1843																		
Sodium	589.592																			
Strontium	407.771																			
Thallium	190.601		4.82122																	
Tin	189.927	-2.11762																		
Titanium	314.946	N/A																		
Vanadium	192.395	0.58185	N/A																	
Zinc	206.200																			

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SOP Name: Inductively Coupled Plasma Atomic Emission Spectroscopy
Perkin Elmer OPTIMA-3000/3300DV/5300DV
SW-846 Method 6010C
SOP Number: **GR-01-100**

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Revision Number: 5.9

Date Revised: 7/22/11
Date Initiated: 4/4/96

Attachment 23.4
Table 3
IFA 1 Working Solution

Element	Concentration (mg/L)
Aluminum	100.0
Calcium	100.0
Iron	40.0
Magnesium	100.0

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SOP Name: Inductively Coupled Plasma Atomic Emission Spectroscopy
Perkin Elmer OPTIMA-3000/3300DV/5300DV
SW-846 Method 6010C
SOP Number: **GR-01-100**

Revision Number: 5.9

Date Revised: 7/22/11
Date Initiated: 4/4/96

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Attachment 23.5
Table 4
IFA 2 Working Solution

Element	Concentration (mg/L)
Chromium	20.0
Copper	20.0
Manganese	20.0
Nickel	20.0
Titanium	20.0
Vanadium	20.0

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Attachment 23.6
Table 5
Optima Working Standard Concentrations (mg/L)

Element	Standard 4	Standard 3	Standard 2	Standard 1
Aluminum	5.000	2.500	0.500	0.050
Arsenic	5.000	2.500	0.500	0.100
Barium	1.000	0.500	0.100	0.010
Beryllium	0.100	0.050	0.010	0.001
Boron	1.000	0.500	0.100	0.050
Cadmium	1.000	0.500	0.100	0.005
Calcium	10.000	5.000	1.000	0.500
Chromium	1.000	0.500	0.100	0.010
Cobalt	1.000	0.500	0.100	0.010
Copper	1.000	0.500	0.100	0.010
Iron	1.000	0.500	0.100	0.010
Lead	1.000	0.500	0.100	0.050
Lithium	1.000	0.500	0.100	0.008
Magnesium	10.000	5.000	1.000	0.500
Manganese	1.000	0.500	0.100	0.010
Molybdenum	1.000	0.500	0.100	
Nickel	1.000	0.500	0.100	0.010
Potassium	10.000	5.000	1.000	0.100
Selenium	5.000	2.500	0.500	0.100
Silicon	1.000	2.500	0.500	0.100
Silver	1.000	0.500	0.100	0.005
Sodium	10.000	5.000	1.000	0.500
Strontium	1.000	0.500	0.100	0.050
Vanadium	5.000	2.500	0.500	0.100
Zinc	5.000	2.500	0.500	0.100
Titanium	1.000	0.500	0.100	
Vanadium	1.000	0.500	0.100	0.010
Zinc	1.000	0.500	0.100	0.010

Attachment 23.7
Table 6
IFB-1 Working Solution

Element	Concentration (mg/L)
Aluminum	100.0
Barium	0.5
Beryllium	0.1
Calcium	100.0
Cadmium	1.0
Cobalt	0.5
Chromium	0.5
Copper	0.5
Iron	40.0
Magnesium	100.0
Manganese	0.5
Nickel	1.0
Lead	1.0
Silver	1.0
Vanadium	0.5
Zinc	1.0

Attachment 23.8
Table 7
IFB-2 Working Solution

Element	Concentration (mg/L)
Aluminum	1.0
Arsenic	1.0
Boron	1.0
Calcium	5.0
Chromium	20.0
Copper	20.0
Magnesium	5.0
Manganese	20.0
Molybdenum	1.0
Sodium	5.0
Nickel	20.0
Selenium	1.0
Titanium	20.0
Thallium	1.0
Vanadium	20.0

Attachment 23.9
 Table 8
 Element Conditions for the Optima 3000

Analyte	Wavelength	Background Correction	Lower BGC (nm)	Upper BGC (nm)	Plasma L/min.	Aux. L/min	Neb L/min	Power Watts	Viewing Distance (mm)
Ag	328.068	2-point	-0.02	0.028	15	0.5	0.8	1300	15
Al	396.146	1-point	-0.044		15	0.5	0.8	1300	15
As	188.979	1-point	0.034		15	0.5	0.8	1300	15
B	249.769	1-point	-0.052		15	0.5	0.8	1300	15
Ba	455.403	2-point	-0.1	0.082	15	0.5	0.8	1300	15
Be	234.863	1-point	0.044		15	0.5	0.8	1300	15
Ca	315.874	1-point	-0.023		15	0.5	0.8	1300	15
Cd	214.436	1-point	-0.056		15	0.5	0.8	1300	15
Ce	413.764	2-point	-0.08	0.025	15	0.5	0.8	1300	15
Co	228.616	1-point	0.016		15	0.5	0.8	1300	15
Cr	205.563	1-point	0.033		15	0.5	0.8	1300	15
Cu	324.741	1-point	0.038		15	0.5	0.8	1300	15
Fe	238.204	1-point	-0.024		15	0.5	0.8	1300	15
K	766.49	2-point	-0.03	0.041	15	0.5	0.8	1300	15
Li	670.784	2-point	-0.03	0.08	15	0.5	0.8	1300	15
Mg	279.071	1-point	0.07		15	0.5	0.8	1300	15
Mn	257.81	1-point	0.036		15	0.5	0.8	1300	15
Mo	202.031	2-point	-0.02	0.028	15	0.5	0.8	1300	15
Na	589.582	2-point	-0.07	0.056	15	0.5	0.8	1300	15
Ni	231.604	1-point	0.032		15	0.5	0.8	1300	15
Pb	220.353	2-point	-0.021	0.019	15	0.5	0.8	1300	15
Sb	217.584	1-point	-0.015		15	0.5	0.8	1300	15
Sc (I.S.)	357.274	1-point	0.063		15	0.5	0.8	1300	15
Se	196.036	1-point	-0.019		15	0.5	0.8	1300	15
Si	251.611	2-point	-0.021	0.016	15	0.5	0.8	1300	15
Sn	189.92	2-point	-0.019	0.019	15	0.5	0.8	1300	15
Sr	407.771	2-point	-0.055	0.071	15	0.5	0.8	1300	15
Ta	234.946	1-point	0.07		15	0.5	0.8	1300	15
Ti	190.901	2-point	-0.006	0.023	15	0.5	0.8	1300	15
Tl	292.395	1-point	0.035		15	0.5	0.8	1300	15
Zn	206.2	2-point	-0.074	0.042	15	0.5	0.8	1300	15
Y (I.S.)	371.029	1-point	0.038		15	0.5	0.8	1300	15

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

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**Attachment 23.11
 Table 10
 Spiking Solutions Final Concentrations**

Element	Aqueous Samples (ug/L)	Solid Samples (ug/L)
Aluminum	2000	1250
Arsenic	2000	1250
Barium	400	250
Beryllium	40	25
Boron	400	250
Cadmium	400	250
Calcium	20000	12500
Chromium	400	250
Cobalt	400	250
Copper	400	250
Iron	400	250
Lead	400	250
Lithium	400	250
Magnesium	20000	12500
Manganese	400	250
Molybdenum	400	250
Nickel	400	250
Potassium	20000	12500
Selenium	400	250
Silicon	400	250
Silver	400	250
Sodium	20000	12500
Strontium	400	250
Sulfur	2000	1250
Thallium	2000	1250
Tin	2000	1250
Titanium	400	250
Vanadium	400	250
Zinc	400	250

Attachment 23.12

Table 11

SCV Concentration when Diluted according to Instructions

Element	Aqueous Samples (ug/L)
Aluminum	2500
Arsenic	2500
Barium	500
Beryllium	50
Boron	500
Cadmium	500
Calcium	5000
Chromium	500
Cobalt	50
Copper	500
Iron	500
Lead	500
Lithium	500
Magnesium	5000
Manganese	500
Molybdenum	500
Nickel	500
Potassium	5000
Selenium	2500
Silicon	2500
Silver	500
Sodium	5000
Strontium	500
Sulfur	2500
Thallium	2500
Tin	2500
Titanium	500
Vanadium	500
Zinc	500

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SOP Name: Inductively Coupled Plasma Atomic Emission Spectroscopy
 Perkin Elmer OPTIMA-3000/3300DV/5300DV
 SW-846 Method 6010C
 SOP Number: GR-01-100

Revision Number: 5.9

Date Revised: 7/22/11
 Date Initiated: 4/4/96

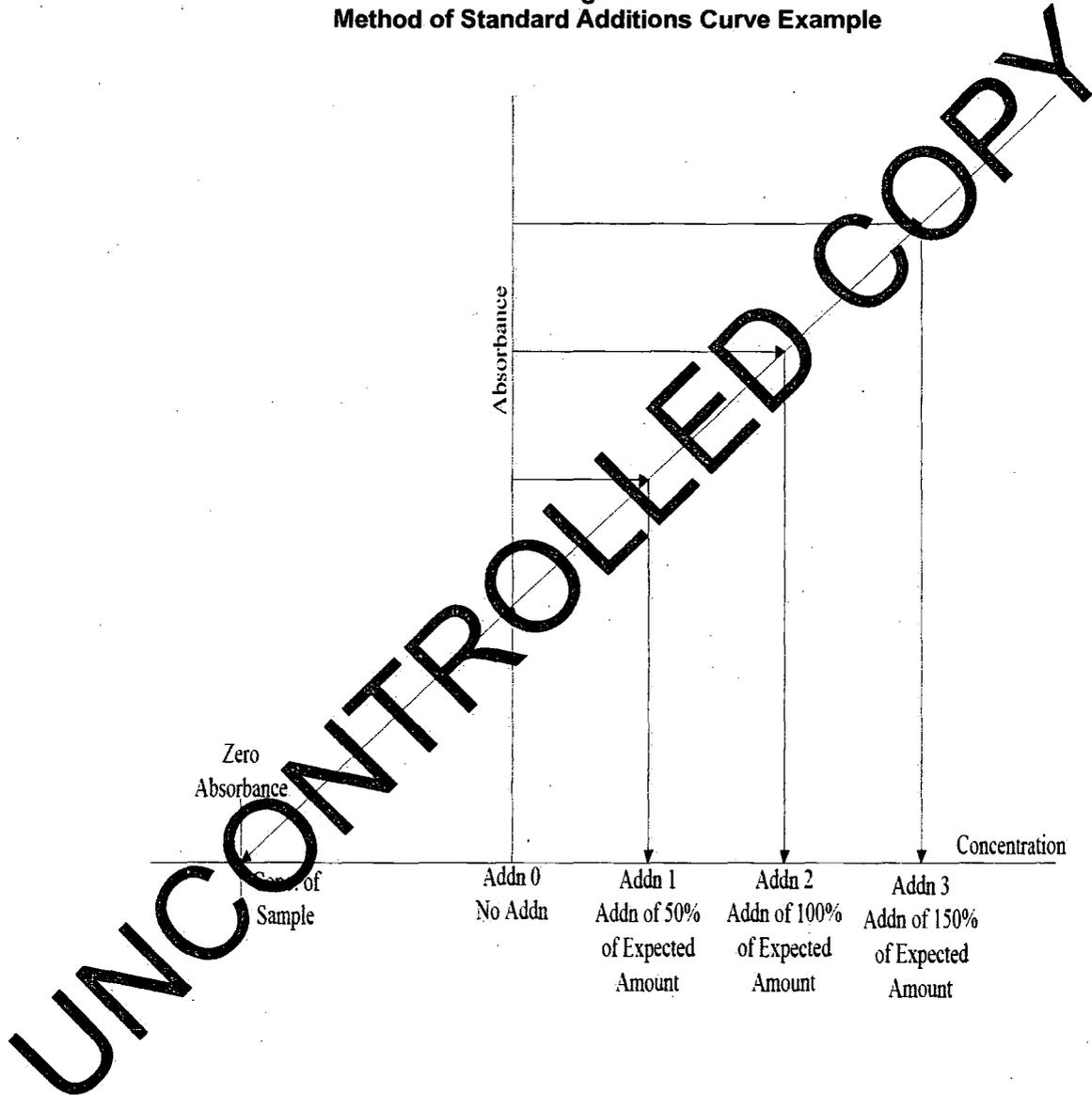
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**Attachment 23.13
 Table 12
 CRL Working Standard Concentrations**

Element	Concentration (ug/L)
Aluminum	100
Arsenic	200
Barium	20
Beryllium	2
Boron	100
Cadmium	10
Calcium	1000
Chromium	20
Cobalt	20
Copper	20
Iron	20
Lead	100
Lithium	10
Magnesium	1000
Manganese	20
Niobium	200
Nickel	20
Potassium	200
Selenium	200
Silicon	200
Silver	10
Sodium	1000
Strontium	100
Sulfur	200
Thallium	200
Tin	200
Titanium	200
Vanadium	20
Zinc	20

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Attachment 23.14
Figure 2
Method of Standard Additions Curve Example



Addendum 23.15
Generating Inter-Element Correction Factors (IEC) For ICP

1) Establishing Background Points.

- a) Perform a normal instrument calibration for all of ICP target analytes, using multi-element calibration standards.
- b) Analyze each metal from a single element standard as a sample, making sure the standard concentration is at the top of the linear range for each analyte. Be certain to use a very long rinse cycle between elements (nominally 20 minutes). A shorter cycle time may be used if there is no carry over.
- c) When the analysis is complete, review the blank spectra, lowest standard spectra and each single element spectra. Find the point on either side of each spectrum that has no response (baseline response) or the point that has the smallest number of peaks. If one side of the peak has multiple responses or peaks making it difficult to select a point, then do not establish a background point on that side. Instead, use the one background point from the opposite side. (Refer to pages 6, 7 and 8).

2) Generation of IEC Factors.

- a) Perform a normal instrument calibration for all target analytes.
- b) Analyze 5 replicates for each major interference standard (Al, Ca, Fe and Mg) at the same concentration as in 1(b) above.
- c) Perform a 20 minute rinse between each analysis to prevent any potential analyte carryover.
- d) The 5 replicates will verify that the previous analyzed element is completely rinsed out prior to each analysis. For example, if running Al then Ca, look for a decreasing emission response for Al during the Ca replicate analyses. If there is some carryover, then repeat the 5 replicate analyses for that analyte. The long rinse is very important in preventing carryover from the previous metal.
- e) Method 6010 requires at a minimum that IEC factors for Al, Ca, Fe and Mg be calculated. The methods also state that IEC factors must be generated for any other metals that may impact data.
- f) Creating IEC factors for Ce, Cd, Cr, Cu, Co, Mn, Mo, Ni, Ti and V in addition to the method required analytes improves the ICP analysis.
- g) Once the preceding metals have been analyzed, run the software program to generate correction factors (manufacturer and instrument specific).

3) Validation of Newly Established IEC Factors

- a) After correction factors have been generated and properly stored in the instrument, perform a test to verify their performance. Remember to change the regular method to use the same background points as the method used to generate new IEC factors. Also, make sure method IEC factors are referenced or selected as part of the method calculations.
- b) Perform a normal instrument calibration and analyze a set of initial quality control samples. These initial samples must include two interference check samples (CLP-designated ICS-A, and ICS-AB solutions) prepared in accordance to the interference table in method 6010. Refer to TriMatrix SOP GR-01-100, which describes the preparation, analysis and acceptance for these two QC standards. In the procedure and in LIMS, they are identified as IFA1 and IFA2 (ICS-A), and IFB1 and IFB2 (ICS-AB).
- c) The IFA1 standard contains Al, Ca, Fe, and Mg while the IFA2 solution contains Cr, Cu, Mn, Ni, Ti and V. The IFB1 Standard contains the major interfering elements and all known analytes affected by these major elements. The second solution IFB2 contains all the minor interfering analytes and all known analytes affected by these minor elements.
- d) If new correction factors are working properly then all unspiked analytes in both solutions should pass criteria of ≥ 2 times the reporting limit (RL) and all spiked analytes should pass 80 to 120% of the prepared value.

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Addendum 23.15
Generating Inter-Element Correction Factors (IEC) for ICP

ICP INTERFERENCE CHECK SAMPLE

USEPA-6010C

Laboratory: TriMatrix Laboratories, Inc.

SDG:

Client: Environmental Resource Associates

Project: Semi-Annual Solid PB Study

Sequence: OB18005

Instrument ID: 311

Calibration: UNASSIGNED

Lab Sample ID	Analyte	True	Found	%R	Units
OB18005-IFA1	Aluminum	100	98.5	98.5	mg/L
	Arsenic	0.00	-0.0212		mg/L
	Barium	0.00	-0.00873		mg/L
	Beryllium	0.00	0.000068		mg/L
	Boron	0.00	0.00587		mg/L
	Cadmium	0.00	-0.000000		mg/L
	Calcium	100	96.9	97	mg/L
	Chromium	0.00	-0.00356		mg/L
	Cobalt	0.00	-0.00254		mg/L
	Copper	0.00	-0.00607		mg/L
	Iron	40.0	38.9	97	mg/L
	Lead	0.00	0.0191		mg/L
	Manganese	0.00	-0.000817		mg/L
	Molybdenum	0.00	0.00601		mg/L
	Nickel	0.00	-0.00341		mg/L
	Potassium	0.00	-0.00941		mg/L
	Selenium	0.00	0.0882		mg/L
	Silver	0.00	-0.0196		mg/L
	Sodium	0.00	-0.0250		mg/L
	Strontium	0.00	-0.000342		mg/L
Thallium	0.00	-0.0310		mg/L	
Titanium	0.00	-0.00124		mg/L	
Vanadium	0.00	-0.00527		mg/L	
Zinc	0.00	-0.00511		mg/L	
OB18005-IFA2	Aluminum	0.00	0.0617		mg/L
	Arsenic	0.00	-0.0183		mg/L
	Barium	0.00	-0.00101		mg/L
	Beryllium	0.00	-0.0000118		mg/L
	Boron	0.00	0.0186		mg/L

SOP Name: Inductively Coupled Plasma Atomic Emission Spectroscopy
 Perkin Elmer OPTIMA-3000/3300DV/5300DV
 SW-846 Method 6010C
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Revision Number: 5.9

Date Revised: 7/22/11
 Date Initiated: 4/4/96

Addendum 23.15
Generating Inter-Element Correction Factors (IEC) For ICP

ICP INTERFERENCE CHECK SAMPLE
USEPA-6010C

Laboratory: TriMatrix Laboratories, Inc.
 Client: Environmental Resource Associates
 Sequence: OB18005

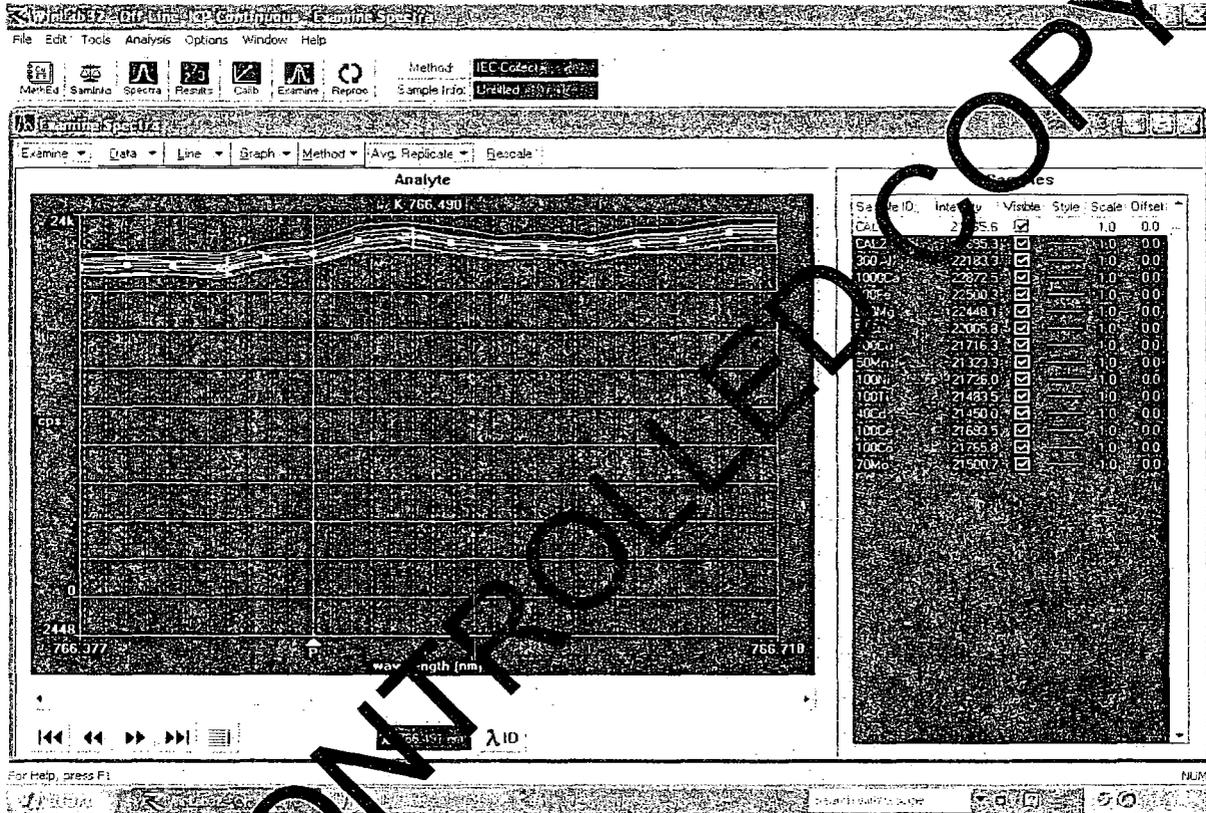
SDG:
 Project: Semi-Annual Solid PE Stud
 Instrument ID: 311
 Calibration: UNASSIGNED

Lab Sample ID	Analyte	True	Found	R	Units
OB18005-IFB1	Manganese	0.500	0.498		mg/L
	Molybdenum	0.00	0.00615		mg/L
	Nickel	1.00	0.975	97	mg/L
	Silver	1.00	0.99	100	mg/L
	Vanadium	0.500	0.500	100	mg/L
	Zinc	1.00	0.96	96	mg/L
OB18005-IFB2	Aluminum	1.00	1.07	107	mg/L
	Arsenic	1.00	1.06	106	mg/L
	Boron	1.00	0.985	98	mg/L
	Calcium		4.98	100	mg/L
	Chromium	20.0	20.3	101	mg/L
	Copper	20.0	21.1	105	mg/L
	Manganese	20.0	19.9	100	mg/L
	Molybdenum	1.00	1.00	100	mg/L
	Nickel	20.0	20.6	103	mg/L
	Selenium	1.00	1.01	101	mg/L
	Vanadium	5.00	5.16	103	mg/L
	Thallium	1.00	0.885	88	mg/L
	Titanium	20.0	20.1	101	mg/L
	Vanadium	20.0	20.5	102	mg/L

* Values outside of QC limits

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Addendum 23.15
Generating Inter-Element Correction Factors (IEC) for ICP



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SOP Name: Inductively Coupled Plasma Atomic Emission Spectroscopy
 Perkin Elmer OPTIMA-3000/3300DV/5300DV
 SW-846 Method 6010C

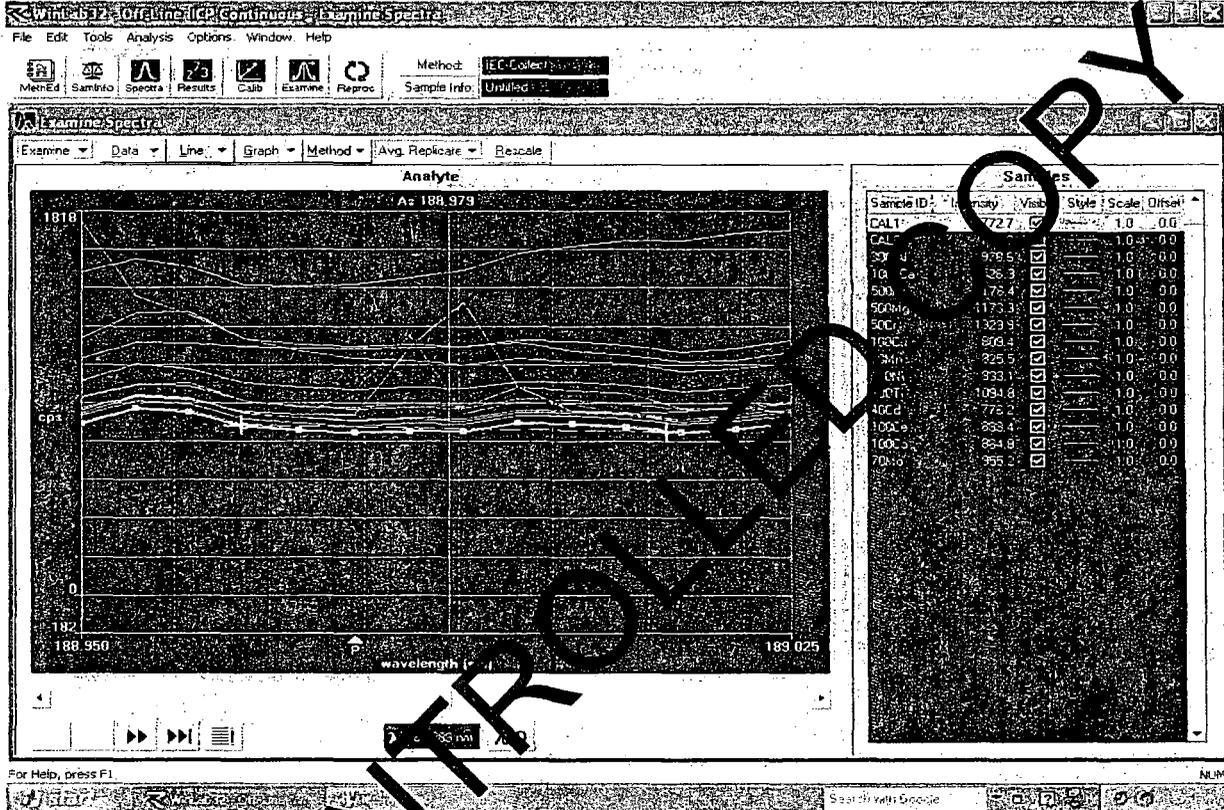
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Generating Inter-Element Correction Factors (IEC) for ICP

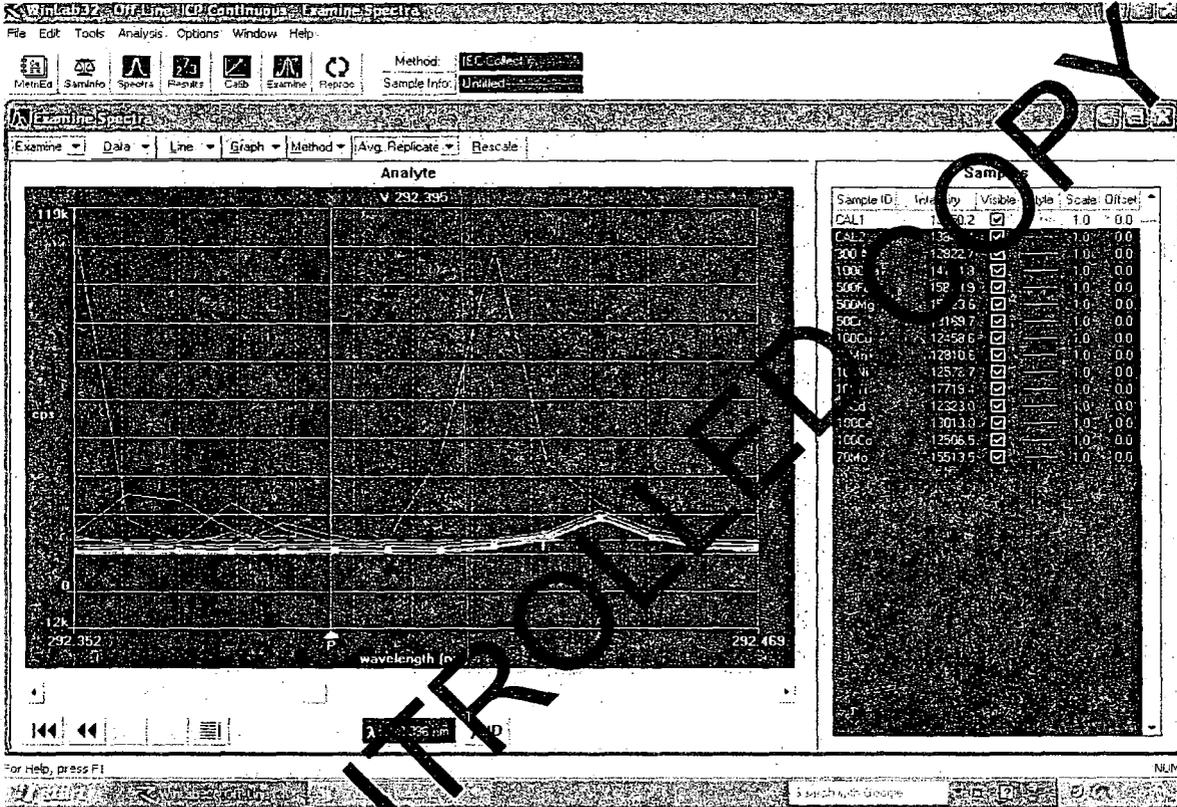


Addendum 23.15 Generating Inter-Element Correction Factors (IEC) for ICP

SOP Name: Inductively Coupled Plasma Atomic Emission Spectroscopy
Perkin Elmer OPTIMA-3000/3300DV/5300DV
SW-846 Method 6010C
SOP Number: GR-01-100

Revision Number: 5.9

Date Revised: 7/22/11
Date Initiated: 4/4/96



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STANDARD OPERATING PROCEDURE
Toxicity Characteristic Leaching Procedure (TCLP)

SW-846 Method 1311

APPROVALS:

Area Supervisor: Marge A. Scott
Marge A. Scott

Date: 07/23/12

QA Officer: John C. Boocher
John C. Boocher

Date: 7-26-12

Laboratory President: Douglas E. Kriscunas
Douglas E. Kriscunas

Date: 7-26-12

Procedure Number: GR-01-119
Revision Number: 2.4

Date Initiated: 7/01/94
Effective Date: 8/10/12

Date Revised: 7/23/12
Pages Revised: All

By: Marge A. Scott
Total Number of Pages: 28

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
_____	_____	_____
_____	_____	_____
_____	_____	_____

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1.0 SCOPE AND APPLICATION

- 1.1 This procedure determines the mobility of specific organic and inorganic analyte in liquid, solid, and multiphase waste by extraction with an acetic acid solution.
- 1.2 A TCLP need not be run if analytes are not detected or are detected but calculated to be below the regulatory limit. This determination must be based on a total compound analysis of the sample.
- 1.3 If analysis of any liquid fraction of a TCLP extract indicates a concentration equal to or greater than the regulatory limit, the waste is hazardous by association and analysis of the remaining fractions is not required. This determination applies to multiphase extracts.
- 1.4 If the bottle tumbler concentration of a volatile analyte exceeds the regulatory limit, the waste is hazardous and ZHE extraction is not required. However, bottle extraction cannot demonstrate any volatile analyte concentration is below the regulatory limit. ZHE must be performed for that purpose.
- 1.5 This procedure is applied to TCLP analytes only, except by client request.
- 1.6 Oily waste samples that filter are assumed to be 100% liquid (filterable), and a total analysis is performed to determine TCLP results (refer to Attachment 20.6 - US Environmental Protection Agency Memorandum #35 dated 06/12/92, page 10). Oily waste and paint waste samples that do not filter are assumed to be 100% solid (nonfilterable) even if appearing to be liquid and a TCLP extraction is performed.

2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 0, July, 1992, Method 1311, "Toxicity Characteristic Leaching Procedure"*

3.0 SUMMARY OF PROCEDURE

- 3.1 For filterable waste containing less than 0.5% dry solids, the filtrate becomes the TCLP extract analyzed. A 0.7 μ m glass fiber filter is used for all TCLP filtrations.
- 3.2 For filterable waste containing greater than or equal to 0.5% solids, an aliquot is filtered and the filtrate stored while the solid phase (after any particle size reduction) is extracted in a rotary tumbler. An extraction fluid volume equal to 20 times the mass of the solid phase is used. One of two extraction fluids are employed, based on the solid phase alkalinity. A special zero headspace extractor (ZHE) is used when extracting volatile TCLP analytes. After the solid phase has tumbled for 18 ± 2 hours, the extraction fluid is separated from the solid phase by filtration.
- 3.3 If multiple phases will not form when mixed, the initial waste filtrate is added to the extraction fluid filtrate, which becomes the TCLP extract analyzed. If incompatible, the two filtrates are analyzed separately. Results of the two analyses are reported as a volume-weighted average concentration in mg/L (refer to Attachment 20.7).

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to Attachment 20.1 for parameters and analytes associated with TCLP regulatory limits.

5.0 REFERENCED STANDARD OPERATING PROCEDURES

- 5.1 TriMatrix Laboratories SOP GR-15-102, *Laboratory Waste Disposal*, latest revision
- 5.2 TriMatrix Laboratories SOP GR-09-104, *Waste Dilution for Semi-Volatile BNA Compounds*, latest revision
- 5.3 TriMatrix Laboratories SOP GR-04-103, *Semi-volatiles Gas Chromatography Mass Spectrometry(GC/MS) Base Neutral Acids(BNA)*, latest revision
- 5.4 TriMatrix Laboratories SOP GR-03-126, *Chlorinated Herbicides by Capillary Column Gas chromatography*, latest revision
- 5.5 TriMatrix Laboratories SOP GR-09-113, *Extraction of Chlorinated Herbicides from Water and Wastewater*, latest revision
- 5.6 TriMatrix Laboratories SOP GR-09-101, *Extraction Base Neutrals Acids(BNA) from Water*, latest revision
- 5.7 TriMatrix Laboratories SOP GR-09-107, *Extraction Organochlorine Pesticides and PCBs from Water*, latest revision
- 5.8 TriMatrix Laboratories SOP GR-03-120, *Gas Chromatography(GC) Organochlorine Pesticides*, latest revision
- 5.9 TriMatrix Laboratories SOP GR-01-147, *Acid Digestion of Total Metals in Aqueous Samples by Block Digestion for TSP*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

- 6.1 Potential interferences that may be encountered during analysis are discussed in individual analytical standard operating procedures.
- 6.2 This is an empirical procedure and must be consistently performed without modification, to attain precise and accurate results.

7.0 SAFETY PRECAUTIONS

- 7.1 Refer to the TriMatrix Chemical Hygiene Plan and Laboratory Safety Manual for routine precautions and safety instructions.
- 7.2 Gloves and approved safety glasses must be worn at all times when handling acids and working in the TCLP laboratory. A plastic apron must also be worn when handling large volumes of

concentrated acid. Concentrated acetic acid fumes are a lung irritant. A Fume hood must be used when handling. Rinse all pipette tips and beakers before disposal or cleaning.

CAUTION: Be certain TCLP tumbler vessels are well vented before tumbling. Each jar must be checked periodically for internal pressure and leakage. Tumbler vessels must be vented cautiously to prevent injury or explosive sample loss. Venting must be performed in a hood.

- 7.3 If a jar leaks or breaks while tumbling, notify the laboratory supervisor and clean up the spill. To prevent a cataclysmic spill that contaminates the tumbler and/or the tumbling room, encase each jar in a plastic bag. However, when a leak does occur (even if contained within the bag), the leachate is compromised and must be repeated.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

- 8.1 For metals and semivolatiles TCLP extractions, at least 100 g sample aliquots are required. For volatiles TCLP extractions, a minimum of 25 g is required. These minimum requirements are based on samples with no filterable liquids. At least 100 g is also needed for a percent solids determination, except for samples with no filterable liquids.
- 8.2 Larger sample aliquots must be extracted based on lower percent solids. Enough extract must be generated to support all required analyses. For multiphase extracts, enough volume must be generated for each phase.
- 8.3 For fibrous materials like cellulose cloth or other relatively low density waste, the sample size collected must be greater to meet the minimum mass required for extraction.
- 8.4 Sample preservation is not required for TCLP samples, but refrigeration may be used if the low temperature will not initiate irreversible physical changes to sample integrity.
- 8.5 TCLP samples for volatiles analysis must be collected and stored in PTFE-lined capped jars, and stored at 0 - 6° C until extraction. Volatiles samples must only be opened immediately before extracting.
- 8.6 TCLP extract preservation is addressed in section 13.10.8.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

- 9.1 The TCLP extractor rotates extraction vessels end-over-end at 30 ± 2 rpm. Five extractors and three zeroheadspace extractors are located in a room adjacent to the organic prep laboratory. This room is monitored and has an ambient temperature of $23 \pm 2^\circ$ C.
- 9.2 Zero-Headspace Extraction Vessels
- 9.2.1 Zero-headspace extraction (ZHE) vessels are used only when waste is tested for volatile analyte mobility. A ZHE is designed to perform liquid phase/solid phase separation, the introduction of extraction fluid, the TCLP extraction and the extract

filtration without opening. The ZHE has an internal volume of 500 mL to 600 mL, and is equipped to accommodate a 90-110 mm, 0.7 μ m glass fiber filter.

9.2.2 The piston within a ZHE must move with no higher than 15 psi. If not, the Viton o-rings within the vessel must be replaced (Millipore or equivalent). If replacing o-rings does not decrease the pressure needed to move the piston, the ZHE must be repaired by the manufacturer before using or be replaced.

9.2.3 A ZHE must be tested for leaks after every extraction by pressurizing to 50 psi and letting sit unattended for one hour. If pressure is lost check all fittings and inspect or replace o-rings. Repeat the leak test. If leakage problems cannot be solved, the ZHE must be repaired by the manufacturer before using or replaced.

9.3 Bottle Extraction Vessels

9.3.1 Bottle extraction jars are used for TCLP metals and semi-volatile extractions. Jars with sufficient volume to hold sample solids and extraction fluid must be used. Headspace is not an issue. Jars used must be inert to the waste being extracted however.

9.3.2 Soda lime flint glass jars are used when evaluating metals and semi-volatiles mobility from the same extraction.

9.3.3 High density polyethylene (HDPE) jars are used when extracting only TCLP metals.

9.4 Filtration Equipment

9.4.1 Filtration equipment must be inert to the waste being extracted. If using PTFE-coated stainless steel, the coating must be unbroken.

9.4.2 All filtrations MUST be performed in a hood.

9.4.3 When waste is evaluated for volatiles, the ZHE vessel is used for filtration. A ZHE supports and holds a 0.7 μ m glass fiber filter. The vessel is designed to withstand up to 50 psi without rupturing the filter.

Note If a filter ruptures, a gas-tight syringe and syringe filter must be used to remove TCLP filtrate and store in a Tedlar[®] bag.

9.4.4 A positive pressure filter holder is used when waste is evaluated for metals and semivolatiles. The holder has an internal capacity of 1.5 L, accommodates a 142 mm diameter 0.7 μ m glass fiber filter, and is capable of exerting 50 psi or greater. Vacuum filtration is not used.

9.4.5 Assemble and operate pressure filters and ZHE vessels per manufacturer's instructions.

9.5 Borosilicate glass fiber filters not containing binder material, with an effective pore size of 0.7 μ m (Millipore or equivalent) are used for all TCLP filtrations. Prefilters must not be used. TCLP filters are fragile and must be handled with care.

- 9.5.1 When evaluating metals mobility, filters must be acid-washed before using, by washing with 1N nitric acid followed by three consecutive washes with deionized distilled water (a minimum of 1 L per rinse).
- 9.6 The pH meter used must be accurate to ± 0.05 units at 25° C.
- 9.7 A Tedlar® bag or 500 mL glass gas-tight syringe must be used to collect any initial liquid phase and final TCLP extract when using ZHE, according to the following scenario:
- 9.7.1 If a waste contains an aqueous liquid phase and less than 1% filterable nonaqueous liquid, a Tedlar® bag or 500 mL glass gas-tight syringe is used to collect and combine the initial filtrate and TCLP extract.
- 9.7.2 If a waste contains greater than 1% nonaqueous filterable liquid (such as oil) in the initial liquid phase, only a Tedlar® bag is used to collect and combine the initial solid/liquid separation and the final extraction.
- 9.7.3 If a waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), a Tedlar® bag is used.
- 9.8 A ZHE extraction fluid transfer device is used to transfer extraction fluid into the ZHE without changing the nature of the extraction fluid.
- 9.9 The laboratory balance used is accurate to within ± 0.01 g.
- 9.10 Beaker and/or Erlenmeyer flask, glass, or Pyrex, 500 mL.
- 9.11 Watchglass, glass. Appropriate diameter to cover breaker or Erlenmeyer flask.
- 9.12 9.5 mm (0.375 inch) standard sieve
- 10.0 ROUTINE PREVENTIVE MAINTENANCE**
- 10.1 TCLP Filtration Devices
- 10.1.1 Change PTFE and rubber gaskets as needed.
- 10.1.2 Frequently examine the PTFE coating on interior surfaces of each ZHE. Scored or abraded surfaces inhibit effective cleaning of the device. If scored or abraded, return to the manufacturer for recoating.
- 10.2 ZHE Devices
- 10.2.1 Replace Viton O-rings before each extraction.
- 10.2.2 Replace worn gaskets as needed.
- 10.2.3 Maintain pressure gauges and replace as needed.

10.3 Rotary Tumblers

10.3.1 Monitor and record revolutions per minute (rpm) for each rotary tumbler. Tumbler velocity must be 30 ± 2 rpm. If velocity exceeds 30 ± 2 rpm perform maintenance and repair of the motor and/or bearings.

11.0 CHEMICALS AND REAGENTS

11.1 Trace metal grade acids must be used. All reagents must conform to specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available.

11.2 Reagent Water: For nonvolatiles extractions, ASTM Type II water or equivalent must be used. For volatiles extractions, the reagent water must also be volatile free.

11.3 Hydrochloric acid (HCl): 1.0 N

11.4 Nitric acid (HNO₃): 1.0 N

11.5 Sodium hydroxide (NaOH): 1.0 N

11.6 Glacial acetic acid(CH₃CH₂OOH)

11.7 Extraction Fluids:

11.7.1 Extraction fluid #1: Add 285 mL glacial CH₃CH₂OOH to ~25 L of reagent water, add 3.215 L of 1.0 N NaOH, and dilute to 50 L. When correctly prepared, the pH of this fluid will be 4.83 ± 0.05 .

11.7.2 Extraction fluid #2: Dilute 285 mL glacial CH₃CH₂OOH with reagent water to a volume of 50 liters. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05 .

Note: Extraction fluid pH must be checked initially to verify the fluids have been made correctly, and before each extraction use. Additionally, both extraction fluids must be monitored for impurities by preparing method performance blanks. If contamination is found or pH is not within specification, the fluid must be discarded and prepared again. These checks must be documented by recording appropriately.

12.0 STANDARDS PREPARATION

12.1 TCLP extracts for metals, designated as Laboratory Control Samples (LCS) or Matrix Spikes (MS) must be spiked before preservation according to the following:

Analyte	Stock (ug/mL)	ICP Analysis		
		Spike added (mL)	Sample Volume (mL)	Final Spike Conc. (ug/mL)

ICP Analysis				
Analyte	Stock (ug/mL)	Spike added (mL)	Sample Volume (mL)	Final Spike Conc. (ug/mL)
Arsenic	500	1.00	100	5.00
Barium	50	1.00	100	0.50
Cadmium	50	1.00	100	0.50
Chromium	50	1.00	100	0.50
Copper*	50	1.00	100	0.50
Lead	50	1.00	100	0.50
Selenium	500	1.00	100	5.00
Silver	50	1.00	100	0.50
Zinc*	50	1.00	100	0.50

ICP/MS Analysis				
Analyte	Stock (ug/mL)	Spike added (mL)	Sample Volume (mL)	Final Spike Conc. (ug/mL)
Arsenic	5.0	1.00	100	0.050
Barium	5.0	1.00	100	0.050
Cadmium	5.0	1.00	100	0.050
Chromium	5.0	1.00	100	0.050
Copper*	5.0	1.00	100	0.050
Lead	5.0	1.00	100	0.050
Selenium	5.0	1.00	100	0.050
Silver	5.0	1.00	100	0.050
Zinc*	5.0	1.00	100	0.050

CVAA Analysis				
Analyte	Stock (ug/mL)	Spike added (mL)	Sample Volume (mL)	Final Spike Conc. (ug/mL)
Mercury	0.100	0.200	4.00	0.005

* Additional State of Michigan regulated metals

12.2 Additional elements may be spiked/analyzed by client request.

13.0 ANALYTICAL PROCEDURE

13.1 Perform preliminary TCLP evaluations on a minimum 100 gram sample aliquot. This aliquot does not undergo TCLP extraction.

13.2 Preliminary evaluations include:

13.2.1 Determination of percent solids.

13.2.2 Determination of percent solids to decide whether a waste is extracted or filtered.

- 13.2.3 Determination of whether the solid portion of a waste requires particle size reduction.
- 13.2.4 Determination of which extraction fluid must be used for a nonvolatile TCLP extraction. Use the TCLP Extraction Worksheet (Attachment 20.4) as a step-by-step guide and as a logbook for recording sample evaluation results.
- 13.3 Percent solids (section 2 on the TCLP Extraction Worksheet) are defined as the sample fraction (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.
- 13.3.1 If a sample will obviously yield no liquid when subjected to pressure filtration (is 100% solids) proceed to section 13.4.
- 13.3.2 If a sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the pressure filter holder described in section 9.4.4 and is outlined below.
- 13.3.2.1 Pre-weigh the filter disc and container receiving the filtrate.
- 13.3.2.2 Assemble the filter holder and filter by placing the filter on the support screen and securing.
- 13.3.2.3 Weigh out a sample aliquot (100 g minimum) and record the mass.
- 13.3.2.4 Let slurries stand to permit the solid phase to settle out before filtering. Wastes that settle slowly may be centrifuged. However, centrifugation must be used only as a filtration aid. If used, decant any liquid into the filter before adding the solid phase. Filter the entire aliquot by making a quantitative transfer into the filter holder but do not rinse.
- 13.3.2.5 Spread unfilterable solids evenly over the surface of the filter. Filtration must be at room temperature to prevent viscosity changes from affecting results.
- Note:** If greater than 1% of the original mass obviously adheres to the transfer container, determine the mass of this residue and subtract it from the original aliquot mass recorded. This subtraction determines the actual mass transferred.
- 13.3.2.6 Gradually apply gentle pressure up to 10 psi, until pressurized air moves through the filter. If no air moves through after being at 10 psi for 2 minutes, and if no additional liquid passes through the filter, slowly increase the pressure in 10 psi increments at 2 minute intervals, to a maximum of 50 psi. When air begins to move through the filter or when liquid flow ceases after 2 minutes at 50 psi, stop the filtration.
- Note:** Instantaneous application of pressure degrades the glass fiber filter and causes premature plugging. Pressure increases must be gradual. Especially below 10 psi.

13.3.2.7 Any sample remaining on the filter is defined as the solid phase which must be TCLP extracted. The liquid filtrate is the initial liquid phase.

Note: Certain oily waste and paint waste, may contain liquid material that will not filter, even after pressure filtration to 50 psi. Such unfilterable liquid is a TCLP solid and must be extracted. No attempt must be made to continue filtering by using more than one filter disc or higher pressure.

13.3.2.8 Determine liquid phase mass by subtracting the filtrate container mass from the total mass of the filtrate/container. Determine the solid phase mass by subtracting the liquid phase mass from the waste aliquot used. Record the liquid and solid phase masses. Calculate percent solids as follows:

$$\text{Percent Solids} = (M_s / M_w) * 100$$

Where:

M_s = solid phase mass in grams
 M_w = waste aliquot mass in grams

13.4 Calculation of percent solids must be done on dry solids:

13.4.1 To dry a solid phase, remove the solid phase and filter from the filtration apparatus.

13.4.2 Dry the filter and solid phase at $100 \pm 20^\circ \text{C}$ until two successive weighings yield the same value within $\pm 1\%$. Record the final dry mass.

Note: Caution must be taken to ensure a solid phase will not flash when heated. The drying oven must be vented to a hood.

13.4.3 Calculate percent dry solids as follows:

$$S_d = (M_{w+f} - M_f) * 100 / M_w$$

Where:

M_{w+f} = dry waste+filter mass (g)
 M_f = tared filter mass (g)
 M_w = total waste aliquot mass (g)
 S_d = Dry Solids (%)

13.5 When percent dry solids are less than 0.5%, proceed to section 13.10 if a nonvolatiles extraction is required. Proceed to section 13.11 if a volatiles TCLP is required. If solids are equal to or greater than 0.5%, use a new sample aliquot to determine if particle size reduction is necessary and to determine which extraction fluid is required.

13.6 A filterable oil with apparent solids greater than 0.5% due only to an oily residue on the filter because of premature clogging, is considered 100% liquid and its own leachate. TCLP results will be obtained from the filtered oil only. The oily filter/residue will not be TCLP extracted. Volatiles TCLP results will also be obtained from analysis of the filtered oil only.

13.7 Solid phase particle size reduction:

13.7.1 Particle size reduction is required before TCLP extraction unless a solid has a surface area equal to or greater than $3.1 \text{ cm}^2/\text{g}$ or is smaller than 1 cm in its narrowest dimension. It must be capable of passing through a 9.5 mm standard sieve.

13.7.2 If a solid phase will not pass through the sieve, reduce particle size by crushing, cutting, or grinding until it does. Record all steps taken to reduce particle size on the TCLP Extraction Worksheet in sections 2.3, 3.2, and 4.4.

Note: Surface area criteria are meant for filamentous paper, cloth, and similar waste materials. Actual measurement of surface area after particle size reduction is not required, nor is it recommended.

13.8 If a solid phase is greater than or equal to 0.5% and will be extracted for nonvolatiles, determine the appropriate extraction fluid as follows (complete Section 3 on the TCLP Extraction Worksheet):

Note: TCLP extraction for volatile constituents uses only extraction fluid #1. If TCLP extraction for nonvolatiles is not required, proceed to section 13.11.

13.8.1 Reduce a small subsample of solid phase to particles of approximately 1 mm in diameter or less. Weigh 5.0 g into 500 mL beaker or Erlenmeyer flask.

13.8.2 Add 96.5 mL of reagent water, cover with a watchglass and record the temperature. Stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the initial pH. The pH must be taken at room temperature (25°C). If the initial pH is less than 5.0, use Extraction Fluid #1. Proceed to section 13.10.

13.8.3 If pH is greater than 5.0, add 3.5 mL 1N HCl and slurry briefly. Cover with a watchglass and heat at 50°C for 10 minutes.

13.8.4 Let the solution cool to room temperature (25°C) then measure and record the pH. If pH is less than 5.0, use extraction fluid #1. If pH is greater than 5.0, use extraction fluid #2.

13.9 If a sample aliquot used for preliminary evaluation is 100% solid, then it can be used for nonvolatiles TCLP extraction (assuming at least 100 g remain). The aliquot used for percent solids determination is also appropriate for nonvolatiles extraction if enough solid was obtained to support all analyses required. If an adequate amount remains, proceed to section 13.10. If a sample is not a 100% solid waste, and a solids determination was done, another aliquot must be taken for TCLP volatiles extraction.

13.10 Procedure to use when volatiles are not involved:

13.10.1 For nonvolatiles TCLP extractions, 100 g of sample is required (complete section 4 through 7 on the TCLP Extraction Worksheet). A larger aliquot may be needed depending on percent solids content, on whether any initial liquid phase is miscible with the solid phase extract and on which analytes are required. Enough solid phase extract volume must be obtained to support all analyses requested. If an extract

volume generated by a single extraction is insufficient to support all analyses, another sample aliquot must be extracted and the extracts combined before analysis.

13.10.2 If a waste yields no liquid when subjected to pressure filtration (is 100% TCLP solids), weigh out at least 100 g and proceed to section 13.2.9.

13.10.3 If a waste is liquid or multiphasic, pressure filtration is required as follows:

13.10.3.1 Pre-weigh the container receiving filtrate.

13.10.3.2 Assemble the filter holder and filter. Place the filter on the support screen and secure. Acid-wash the filter if evaluating metals mobility.

Note: Acid-washed filters may be used for any nonvolatile extraction, even when metals are not of concern.

13.10.3.3 Weigh out at least 100 g of waste sample and record the mass. If a waste contains < 0.5% dry solids, the liquid filtrate is defined as the TCLP extract. Enough sample must be filtered so the volume can support all required analyses. For wastes containing $\geq 0.5\%$ dry solids, use the percent solids value obtained earlier to determine the minimum sample mass over 100 g needed to support all TCLP analyses required.

13.10.3.4 Let sedimentary samples settle out before filtering. Wastes that settle slowly may be centrifuged. However, centrifugation must be used only as a filtration aid. If used, decant any liquid into the filter before adding the solid phase. Filter the entire aliquot by making a quantitative transfer into the filter holder, without rinsing.

13.10.3.5 Spread unfilterable solids evenly over the surface of the filter. Filtration must be at room temperature to prevent viscosity changes from affecting results.

Note: If greater than 1% of the original mass obviously adheres to the transfer container, determine the mass of this residue and subtract it from the original aliquot mass recorded. This determines the actual mass transferred to the filter.

13.10.3.6 Gradually apply gentle pressure up to 10 psi, until air moves through the filter. If no gas moves through after being at 10 psi for 2 minutes, and if no additional liquid passes through the filter, slowly increase the pressure in 10 psi increments at 2 minute intervals, to a maximum of 50 psi. When air begins moving through the filter, or when liquid flow ceases after 2 minutes at 50 psi, stop the filtration.

Note: Instantaneous application of pressure degrades the glass fiber filter and causes premature plugging. Pressure increases must be gradual. Especially below 10psi.

13.10.3.7 Waste remaining on the filter is defined as the solid phase which must be TCLP extracted. Any filtrate is defined as the liquid phase. Weigh the

filtrate. The liquid phase is analyzed as the TCLP extract if dry solids are less than 0.5% or stored at 0 - 6° C until the solid phase is extracted, if solids are equal to or greater than 0.5%.

Note: Certain oily waste and paint waste, may contain liquid material that will not filter, even after the pressure filtration described above. Such unfilterable liquid is defined as a TCLP solid and must be extracted. No attempt must be made to continue filtering by using a fresh filter. Use only one filter.

13.10.3.8 If a waste contains < 0.5% dry solids proceed to section 13.10.8. If a waste passes the 9.5 mm sieve as received, quantitatively transfer the solid phase and filter into an extraction bottle, then proceed to section 13.10.4.

13.10.3.9 Prepare solid phases for extraction by crushing, cutting, or grinding to a surface area or particle size that will pass through the 9.5 mm sieve. When surface area or particle size has been reduced, quantitatively transfer into an extraction bottle. Include the filter used to separate the initial liquid from the solid phase.

13.10.4 Determine the volume of extraction fluid to add to the extraction bottle as follows:

$$M_{\text{ext fluid}} = (20/100) * S_d * M_w$$

Where:

- $M_{\text{ext fluid}}$ = Extraction fluid mass needed (g)
- M_w = Total waste aliquot mass (g)
- S_d = Dry Solids (%)

13.10.5 Check the extraction fluid pH and record in the appropriate TCLP Extraction Fluid Logbook (Attachments 20.2 and 20.3). Slowly add extraction fluid to the extraction bottle. Close the lid tightly using PTFE tape to ensure a tight seal. Secure in the tumbler, and rotate at 30 ± 2 rpm for 18 ± 2 hours. The extraction room temperature must be maintained at 23 ± 2° C during the extraction.

Note: While tumbling, pressure may build within extractor bottles containing lime or calcium carbonate waste from carbon dioxide. To relieve excess pressure, bottles must be periodically opened after extracting 15 minutes, 30 minutes and 1 hour. Vent the bottles into a hood.

13.10.6 Following extraction, filter the extract fluid through a new 0.7 µm glass fiber filter, as outlined previously. The filter may be changed if necessary, to speed up filtration. Filters must be acid-washed if evaluating metals mobility.

13.10.7 Complete TCLP extracts as follows:

13.10.7.1 If a waste contains no initial liquid phase, the extraction bottle filtrate is the complete TCLP extract.

- 13.10.7.2 If multiple phases will not result, combine the entire extraction bottle filtrate with the initial liquid phase separated from the waste aliquot used. The combined liquid becomes the complete TCLP extract.
- 13.10.7.3 If an initial liquid phase from the waste does not appear compatible with extraction bottle filtrate, do not combine. Each must be analyzed separately and final TCLP results obtained mathematically.
- 13.10.8 Following collection of a complete extract, the pH must be taken and recorded. Immediately aliquot the extract for each analysis and preserve.
 - 13.10.8.1 Metals aliquots must be acidified with nitric acid to pH ~2. If precipitation is observed when nitric acid is added to a small volume, do not acidify and digest as soon as possible.
 - 13.10.8.2 All aliquots must be stored by refrigeration (0 - 6° C) unless prepared for analysis immediately.
 - 13.10.8.3 Extracts must be prepared and analyzed according to appropriate analytical procedures. Aliquots for metals must be acid digested except when digestion causes analyte loss. If an undigested extract analysis result exceeds a regulatory limit, the waste is hazardous and extract digestion is not required. However, data from undigested extracts cannot be used to demonstrate a waste is not hazardous. If individual phases are analyzed separately, determine each phase volume to ± 0.5%, conduct the appropriate analysis, and combine individual phase results using a volume-weighted average:

Final Analyte

$$\text{Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:

- V₁ = Volume of the first phase (L)
- C₁ = Concentration of analyte in the first phase (mg/L)
- V₂ = Volume of the second phase (L)
- C₂ = Concentration of analyte in the second phase (mg/L)

13.10.9 Report TCLP analyte concentrations with the regulatory limits listed in Attachment 20.1.

13.11 Procedure to use when volatiles are involved:

When TCLP volatiles analysis is required, a ZHE must be used to obtain the TCLP extract. Extracts obtained from a ZHE must not be analyzed for metals, pesticides or base-neutrals. The TCLP/ZHE Worksheet (Attachment 20.5) must be completed when volatiles are extracted.

13.11.1 A ZHE has approximately 500 mL internal capacity. No more than 25 g of solid phase can be extracted, due to adding extraction fluid equal to 20 times the solid's mass. (Only the sample fraction from which no additional liquid is filterable at 50 psi is extracted)

- 13.11.2 Introduce sample into a ZHE for extraction once and do not open until the solid phase extract has been collected. Repeated filling to obtain more extractable solid from a low solids waste is not permitted.
- 13.11.3 Do not expose waste samples, initial liquid phases or extracts to the air for any more time than absolutely necessary. These materials must be handled while cold (0 - 6° C), to minimize volatiles loss.
- 13.11.4 Pre-weigh an evacuated Tedlar® bag and set it aside. Express liquid directly into the bag.
- 13.11.5 Place a ZHE piston within ZHE body (it may be helpful to moisten the piston O-rings with extraction fluid). Adjust the piston to a height that minimizes the piston's movement once the ZHE is charged with sample. Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body. Secure the glass fiber filter between the support screens and set aside. Set the liquid inlet/outlet flange (top flange) aside.
- 13.11.6 If a waste is 100% solids, weigh out no more than 25 g and record the mass.
- 13.11.7 If a waste contains < 0.5% dry solids, the liquid filtrate is the TCLP extract. Weigh out up to 500 g for charging and record the mass.
- 13.11.8 If a waste contains > 0.5% solids, weigh out a calculated sample charging mass up to 500 g and record. Calculate the charging mass to weigh out as follows:
- Sample charging mass (g) = (25*100)/percent solids
- 13.11.8 If solid phase particle size reduction is required, crush, cut, or grind to a surface area or particle size as previously described. The solid phase and reduction equipment must be cooled to 0 - 6° C before performing the reduction. The technique used must not generate heat in and of itself. Air exposure during particle reduction must be avoided to the highest extent possible.
- Note:** Do not sieve due to the possibility that volatiles will be lost. Use a graduated ruler instead.
- 13.11.9 Do not let slurries stand for solid phase settling. Do not centrifuge waste before charging the ZHE.
- 13.11.10 Rapidly and quantitatively transfer the entire sample (all phases) into the ZHE without rinsing. Secure the filter and support screens onto the top flange and secure the top flange to the ZHE body. Tighten all ZHE fittings and place in a vertical position (gas inlet/outlet flange on the bottom).

Note: If greater than 1% of the original sample mass obviously adheres to the transfer container, determine the residue's mass and subtract from the charging mass.

Attach a gas line to the gas inlet/outlet valve (bottom flange), then with the liquid inlet/outlet valve (top flange) open begin applying gentle pressure of 1-15 psi to force all headspace out of the ZHE device. Perform the expulsion in a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If a 0 - 6° C sample temperature inhibits filtration, let the sample warm to room temperature in the ZHE before filtering.

- 13.11.11 Attach the evacuated pre-weighed Tedlar® bag to the liquid inlet/outlet valve, then open the valve. Begin applying gentle pressure of 1-10 psi to force any liquid phase into the bag. If no additional liquid is expelled in any two minute interval, slowly increase the pressure in 10 psi increments or less to a maximum of 50 psi. Continue at the same pressure until no additional liquid is expelled after two minutes, then proceed to the next higher pressure increment. When liquid flow has ceased once 50 psi is reached and no additional filtrate is expelled after two minutes, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, then disconnect and weigh the Tedlar® bag.

Note: Instantaneous application of pressure degrades glass fiber filters and causes premature plugging. Always increase pressure gradually.

- 13.11.12 Particulate material remaining in a ZHE after sample filtration is the solid phase. The filtrate is the liquid phase. Place Tedlar® bags containing liquid phase in the walk-in cooler until the solid phase has been tumbled and the two phases can be combined. All Tedlar® bags containing liquid phase or extract must be labeled.

Note: Some wastes such as oily sludges and paint waste, will obviously contain material that appears to be a liquid. Even after applying maximum pressure, this material may not filter. If such is the case, the material within the ZHE is defined as a TCLP solid and is extracted.

If a waste contains < 0.5% dry solids, the ZHE filtrate is defined as the TCLP extract and is ready for analysis. Otherwise, determine the mass of extraction fluid #1 to add to the ZHE as follows:

$$\text{Extraction fluid \#1 (g)} = (20/100) * \text{percent solids} * \text{sample charging mass (g)}$$

- 13.11.13 Add extraction fluid to the ZHE and tumble to extract, as follows (fluid #1 is used in all cases):

13.11.13.1 With the ZHE in the vertical position, attach a line from the fluid reservoir to the liquid inlet/outlet valve. The line used must be pre-flushed with fluid to eliminate air pockets. Release pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve and pump the calculated amount of extraction fluid into the ZHE.

13.11.13.2 After adding, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure all valves are closed. Manually rotate the ZHE end-over-end 2 or 3 times. Reposition in the vertical position with the liquid inlet/outlet valve on top. Pressurize enough to move the piston (about 5-15 psi) then slowly open the liquid inlet/outlet

valve to bleed any headspace from introducing extraction fluid (do in a hood). The bleeding must be done quickly and stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE to 5 - 10 psi and check all fittings to ensure each is closed.

13.11.13.3 Place in a ZHE rotary tumbler and rotate at 30 ± 2 rpm for 16 ± 2 hours. The extraction room temperature must be monitored and maintained at $23 \pm 2^\circ$ C during extraction.

13.11.14 Following extraction, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas.

13.11.14.1 If no gas release is observed, the extractor has leaked. Check for leaks as specified in section 9.2.3, repeating the extraction with a new sample aliquot after repairs are made.

13.11.14.2 If a ZHE is still pressurized after extraction, the liquid in the extractor is filtered out. If the waste contained a compatible initial liquid filtrate, the solid phase extract is filtered directly into the same Tedlar[®] bag.

13.11.14.3 A separate Tedlar[®] bag must be used if combining the solid phase extract and liquid filtrate forms multiple phases, or if one Tedlar[®] bag will not hold the entire volume of both liquids.

13.11.14.4 Filter extract fluid through the glass fiber filter already in the ZHE by increasing pressure gradually, as discussed previously. All extract fluid must be filtered and collected if a Tedlar[®] bag is used, if an extract is multiphase, or if the waste contains an initial liquid phase.

Note: An in-line glass fiber filter may be used if it is suspected the glass fiber filter in the ZHE has ruptured.

13.12.15 If a sample contains no initial liquid phase, the extract filtrate is the TCLP extract. If a sample contains an initial liquid phase, the extract filtrate and the initial liquid phase filtrate combined are the TCLP extract.

13.12.15 Immediately prepare TCLP extracts for analysis and store with minimal headspace at $0 - 6^\circ$ C until analyzed. Analyze extracts according to the appropriate analytical procedure. If individual phases are not miscible, determine the volume of each phase to $\pm 0.5\%$, analyze both and combine results mathematically as follows:

$$\frac{(C_1)(V_1) + (C_2)(V_2)}{V_1 + V_2}$$

where:

V_1 = Volume of the first phase (L)

C_1 = Concentration of analyte in the first phase (mg/L)

V_2 = Volume of the second phase (L)

C_2 = Concentration of analyte in the second phase (mg/L)

13.12.16 Report TCLP analyte concentrations with the regulatory limits listed in Attachment 20.1.

14.0 DATA REPORTING AND DELIVERABLES

14.1 There are no data reporting or deliverable requirements applicable to this procedure.

15.0 QUALITY ASSURANCE

15.1 Extraction Vessel Blanks:

15.1.1 A minimum of one extraction blank using fluid #1 must be prepared after 20 sample extractions in the same ZHE extractor. There are fourteen extractors labeled A-O and four labeled 1 - 4.

15.1.2 A minimum of one extraction blank must be performed for each extraction fluid #1 batch, for all analytes.

15.1.3 An extraction blank is set up for each new lot of tumbler vessels. The extraction blank is extraction fluid tumbled and filtered as if it were a sample. Analyze the tumbled extraction blank to document the absence of contamination from the tumbler vessels. If contamination is found, the lot must be rejected.

15.1.4 Perform a filtration blank each day semi-volatiles/metals samples are tumbled. Perform the filtration blank with extraction fluid #2. Treat the solution as a tumbled leachate and pass through the filter. Analyze the filtered extraction fluid to document the absence of carryover from previous filtrations. If carryover is found, all associated samples with positive results must be re-extracted and re-filtered. If unable to re-extract/re-filter affected samples, the associated sample results must be qualified and narrated.

15.2 A post-extraction matrix spike must be performed for each waste type (including wastewater treatment sludge, soil, and waste) unless a result exceeds the regulatory limit, and data is used solely to demonstrate a waste is hazardous. At least one matrix spike must be analyzed for each analytical batch. Use matrix spiking concentrations listed in individual volatiles and semi-volatiles analytical procedures, but for metals refer to Section 12.0.

15.2.1 Matrix spikes are added to TCLP extracts before preserving for analysis. Do not spike before the TCLP extraction.

15.2.2 In most cases, extracts must be spiked at regulatory limit concentrations. If analyte concentration is less than half a regulatory limit, spike concentration can be as low as half the analyte concentration but not less than five times the method detection limit. To avoid matrix variability, a matrix spike analysis must be done using the same extract volume as an unspiked analysis.

15.2.3 Matrix spikes monitor procedures used for TCLP analysis, and document matrix interference.

15.2.4 Matrix spike recovery is calculated as follows:

$$\%R = 100 \cdot (X_s - X_u) / K$$

where:

- X_s = Spiked extract concentration (mg/L)
- X_u = Unspiked extract concentration (mg/L)
- K = Spike concentration (mg/L)
- %R = percent recovery

15.3 Samples must undergo TCLP extraction within the following hold times:

Parameter	MAXIMUM SAMPLE HOLD TIMES (DAYS)		
	From: Sample Collection:	From: TCLP extraction	From: preparative extraction
	To: <u>TCLP Extraction</u>	To: <u>Preparative Extraction</u>	To: <u>Analysis</u>
Mercury	28	not applicable	28
Metals, except mercury	180	not applicable	180
Volatiles	14	not applicable	14
Semi-volatiles	14	7	40

If any holding time is exceeded, all results obtained must be considered minimal concentrations. Exceeding a holding time can not establish a waste as nonhazardous when results are below the regulatory limit. However, results obtained out of holding time can still establish a waste characterization as hazardous when concentration is above the regulatory limit.

15.4 TCLP logbook entries must be completed for both volatiles and nonvolatiles/metals during initial set-up/percent solids, extraction fluid and pH determinations. Entries must also be made for all filtration steps such as pH measurements and meter identification number, including times completed.

15.5 If an organics/metals extraction bottle leaks or breaks, and the leachate does not tumble for 18 ±2 hours, the sample must be set up again. If a rotary tumbler is shut off for longer than a few minutes or a ZHE loses all (not some) pressure, the sample must be set up again.

15.6 To generate enough leachate for all nonvolatile organics, metals analysis and QC, three liters of leachate must be prepared. Two liters for base-neutrals, pesticides and herbicides. One liter for metals. For only base-neutrals and metals, two liters of leachate must be prepared where one and a half liters goes for base-neutrals, and a half-liter goes for metals. Leachates for non-volatiles organic analysis must be stored in 4 L jars with PTFE-lined lids.

15.7 Extraction fluid is made in 50 L quantities and given a Batch I.D. number. A TCLP extraction blank must be performed on each batch made.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 There is no quantitative demonstration of capability 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 Consult 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 I, Revision 0, July, 1992, Method 1311, "Toxicity Characteristic Leaching Procedure"*

20.0 ATTACHMENTS

- 20.1 TCLP Regulated Analytes List
- 20.2 TCLP Extraction Fluid Number 1 Logbook
- 20.3 TCLP Extraction Fluid Number 2 Logbook
- 20.4 TCLP Extraction Worksheet
- 20.5 TCLP/ZHE Worksheet
- 20.6 US Environmental Protection Agency Memorandum 35, Pg 10
- 20.7 TCLP 2-Phase Calculation Spreadsheet

**Attachment 20.1
 TCLP Regulated Analytes List**

ANALYTE	CAS NUMBER	REGULATORY LIMIT (mg/L)
VOLATILE ORGANIC COMPOUNDS		
Benzene	71-43-2	0.5
2-Butanone (Methyl Ethyl Ketone)	78-93-3	200
Carbon Tetrachloride	56-23-5	0.5
Chlorobenzene	108-90-7	0.0
Chloroform	67-66-3	5.0
1,2-Dichloroethane	107-06-2	0.6
1,1-Dichloroethane	75-35-4	0.7
Tetrachloroethene	127-18-4	0.7
Trichloroethene	78-01-6	0.5
Vinyl Chloride	75-01-4	0.2
SEMI-VOLATILE ORGANIC COMPOUNDS		
1,4-Dichlorobenzene	106-46-7	7.5
2,4-Dinitrotoluene	121-14-2	0.13
Hexachlorobenzene	118-75-1	0.13
Hexachlorobutadiene	67-83-3	0.5
Hexachloroethane	67-72-1	3.0
2-Methylphenol	95-45-7	200
3-Methylphenol	100-59-4	200
4-Methylphenol	106-44-5	200
Nitrobenzene	98-95-3	2.0
Pentachlorophenol	87-86-5	100
Pyridine	110-86-1	5.0
2,4,5-Trichlorophenol	95-95-4	400
2,4,6-Trichlorophenol	88-06-2	2.0
METALS		
Arsenic *	7440-38-2	5.0
Barium	7440-39-3	100
Chromium	7440-43-9	1.0
Chromium	7440-47-3	5.0
Copper *	7440-50-8	100
Lead	7439-92-1	5.0
Mercury	7439-97-6	0.2
Selenium	7782-49-2	1.0
Silver	7440-22-4	5.0
Zinc *	7440-66-6	500

* additional State of Michigan regulated metals

SOP Name: Toxicity Characteristic Leaching Procedure (TCLP)
 SW-846 Method 1311
 SOP Number: GR-01-119

Revision Number: 2.4
 Date Revised: 7/23/11
 Date Initiated: 7/1/94

Attachment 20.2
 TCLP Extraction Fluid Number 1 Logbook



TCLP Extraction Fluid #1
 pH=4.93±0.05

Batch I.D. _____ Date Made _____ Analyst _____
 Amt./Lot# NaOH Used _____ Amt. Glacial Acetic Acid Used _____
 Amount of Reagent Water Used _____ Total Liter Amt. of Batch _____

Initial pH of Batch _____ pH Meter Used/Date Calibrated _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated

TCLP Extraction Fluid #1
 pH=4.93±0.05

Batch I.D. _____ Date Made _____ Analyst _____
 Amt./Lot# NaOH Used _____ Amt. Glacial Acetic Acid Used _____
 Amount of Reagent Water Used _____ Total Liter Amt. of Batch _____

Initial pH of Batch _____ pH Meter Used/Date Calibrated _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated

TCLP Extraction Fluid #1
 pH=4.93±0.05

Batch I.D. _____ Date Made _____ Analyst _____
 Amt./Lot# NaOH Used _____ Amt. Glacial Acetic Acid Used _____
 Amount of Reagent Water Used _____ Total Liter Amt. of Batch _____

Initial pH of Batch _____ pH Meter Used/Date Calibrated _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated

*If pH is out of the acceptable range, note it and make a new batch of fluid

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SOP Name: Toxicity Characteristic Leaching Procedure (TCLP)
 SW-846 Method 1311
 SOP Number: GR-01-119

page 23 of 28

 Revision Number: 2.4
 Date Revised: 7/23/11
 Date Initiated: 7/1/94

 Attachment 20.3
 TCLP Extraction Fluid Number 2 Logbook

 TCLP Extraction Fluid #2
 pH=2.88±0.05

 Batch I.D. _____ Date Made _____ Analyst _____
 Amount Glacial Acetic Acid Used _____
 Amount of Reagent Water Used _____ Total Liter Amt. of Batch _____

Initial pH of Batch _____ pH Meter Used/Date Calibrated _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated

 TCLP Extraction Fluid #2
 pH=2.88±0.05

 Batch I.D. _____ Date Made _____ Analyst _____
 Amount Glacial Acetic Acid Used _____
 Amount of Reagent Water Used _____ Total Liter Amt. of Batch _____

Initial pH of Batch _____ pH Meter Used/Date Calibrated _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated

 TCLP Extraction Fluid #2
 pH=2.88±0.05

 Batch I.D. _____ Date Made _____ Analyst _____
 Amount Glacial Acetic Acid Used _____
 Amount of Reagent Water Used _____ Total Liter Amt. of Batch _____

Initial pH of Batch _____ pH Meter Used/Date Calibrated _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated

*If pH is out of the acceptable range, note it and make a new batch of fluid

Attachment 20.4
TCLP Extraction Worksheet

TriMatrix
Laboratories, Inc. **TCLP Extraction Worksheet**

Client				
Sample Number				

1.0 Sample Description (Liquid, solid, multi-phasic) _____

% Solids Determination

All samples must undergo % solids determination. Only re-use the solids from this section for the fluid determination. Discard any other solids/filtrates from this section.

21 Analyst/Date	/ /	/ /	/ /	/ /
22				
23				
24 Particle Size Reduction (Y/N)	Y N	Y N	Y N	Y N
25 Actual Sample Weight (100g min.)				
26 Total Volume Filtrate(s)	ml	ml	ml	ml

Label the phases of the same number, A=Aqueous, B=Oil, C=Organic Phase, D= Other Phase. Make sure ALL Lab areas are aware of ALL Multi-phasic samples!

Phase A: Density/Volume	dg / ml	dg / ml	dg / ml	dg / ml
Phase B: Density/Volume	dg / ml	dg / ml	dg / ml	dg / ml
Phase C: Density/Volume	dg / ml	dg / ml	dg / ml	dg / ml
Phase D: Density/Volume	dg / ml	dg / ml	dg / ml	dg / ml

2.7 Weight Of Wet Solids (Filter paper wt. + wet solids) _____

2.8 % Wet Solids ((wt. wet solids- wt. filter paper / act. samp. wt.) x 100) _____

If % wet solids are >0.5%, but < 100%, then use equation: (100/ % wet solids) x 100 to determine the actual amount of sample to set up the TCLP in Section 4.3. Also, do the % dry solids to complete all sample record.

If sample is 100% solid, proceed to Section 4.2. If sample is oily and % wet solids are low, then proceed with the % dry solids.

2.9 Weight of Dry Solids (Wt. of filter paper + dry solids) _____

- Sample must be dried at 100C +/- 20C for two readings within +/- 1%.

1.10 % Dry Solids ((wt. dry solids- wt filter paper/Act. samp. wt.) x 100)	%	%	%	%
--	---	---	---	---

If % dry solids are <0.5%, then the filtrate = the TCLP leachate. Sample is 100% liquid. Bring enough sample for all lab areas (see Section 6.3.4 for actual amounts).

If the sample is oily, the % dry solids are low but not <0.5%, and if there are no visible solids on the filter paper, then the oil filtrate = the TCLP leachate (since there is only oil caught in the filter paper).

Make sure all lab areas receive adequate aliquots of filtrate: Metals- 10 mL vial, Prep - 40 mL vial. PLEASE, record in Narrative (Section 7) that sample was oily and filtrate=TCLP leachate.

Fluid Determination

1.1 Analyst / Date	/ /	/ /	/ /	/ /
1.2 Particle Size Reduction (Y/N). If Yes, explain in Narrative, Section 7	Y N	Y N	Y N	Y N
1.3 Initial Temp(C) (Before stirring 5g of sample/90 mL of 0.1N HCL for 5 minutes)	C	C	C	C
1.4 Initial pH (after 5 minutes of stirring)	pH	pH	pH	pH

If initial pH is <5.0, then use Extraction Fluid 1 and proceed to 3.7. If initial pH is >5.0, then proceed to 3.5.

3.5 Add 3.5 mL of 1N HCL to the sample and water, bring to 50C for 10 minutes, and cool to room temp.

1.6 Final pH of sample (cool solution to room temperature)	pH	pH	pH	pH
--	----	----	----	----

If final pH is <5.0, then use extraction fluid 1.

If final pH is >5.0, then use extraction fluid 2.

3.7 Record extraction fluid used (1 or 2) _____

1.8 pH meter used				
1.9 Date				

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Attachment 20.4 (Cont.)
 TCLP Extraction Worksheet



Sample Number: _____

Sample Size For TCLP Set-up

4.1 For 100% solid sample, weight used (100 g minimum) _____

4.2 If % solids were >0.5% and <100%, then record:

4.2.1 Adjusted amount of sample to yield 100g (from Section 2.1) _____

4.2.2 Actual weight of sample (sample - weight of filter papers) _____

4.2.3 Total Amount of Miscible Filtrate(s) (from Section 2.6) _____

4.3 For 100% liquid sample, record filtrate volumes for all lab areas in Section 6.3.4 _____

4.4 Particle Size Reduction (Y/N) _____

Amount of Extraction Fluid (added by weight) For TCLP Set-up.

5.1 Extraction Fluid (1, 2 or 3) and Batch I.D. _____

5.2 pH of Extraction Fluid _____

5.3 TD = Actual solid weight (from Section 4.1 or 4.2.2) _____

TCLP Extraction Record

6.1 Analyst @ set-up Date (MM-DD-YY) _____

6.1.1 Initial Temperature @ set-up (23±2 C) _____

6.1.2 Tumbler I.D. / RPM (30±2 RPM) _____

6.2 Tumbling Times

6.2.1 Time started _____

6.2.2 Time completed _____

6.3 Analyst @ completion Date (MM-DD-YY) _____

6.3.1 Final Temperature @ completion (23±2 C) _____

6.3.2 Initial miscible phase added back to filtered leachate (Y/N). If Y, explain in section 7.0 _____

6.3.3 Final pH of leachate (Not preserved)

6.3.3.1 pH meter I.D. _____

6.3.3.2 _____

6.3.4 Final Volume of Leachate Collected (ml)

6.3.4.1 Metals Lab (normal aliquot = 1L; min. aliquot = 200 mL) _____

6.3.4.2 Prep Lab (normal aliquot = 500 mL; min. aliquot = 500 mL) _____

6.3.4.3 Inorganic Lab (Check with Supervisor for aliquot amount) _____

6.3.5 Volume of non-miscible phase collected (see Section 2.6)

6.3.5.1 Metals Lab (normal aliquot = 1L; min. aliquot = 200 mL) _____

6.3.5.2 Prep Lab (normal aliquot = 500 mL; min. aliquot = 500 mL) _____

6.3.5.3 Inorganic Lab (Check with Supervisor for aliquot amount) _____

6.3.6 Metals Only:

6.3.6.1 Analysis _____

6.3.6.2 Metals spiked (ml) _____

6.3.7 Preservation (pH < 2). Record actual pH _____

7.0 Narrative _____

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Attachment 20.5
 TCLP/ZHE Worksheet


TCLP/ZHE WORKSHEET

ZHE Identification*	A	B	C	D	E	F	G
Client							
Sample Number							
Date Started							
Time Started							
Analyst (Set Up)							
Room Temp. (C)							
Percent Solids							
Wet Sample Weight (g)							
Residue Weight (g)							
Actual Sample Wt (g)							
Filter Weight (g)							
Filter + Solid Wt. (g) wet							
Grams Filtrate							
% Wet Solids							
Adjusted amount of sample to yield 25 grams (See formula below)							
Extraction Fluid Information							
Extraction Fluid Type							
pH of fluid							
Batch Number							
TCLP/ZHE Set-up							
Solid Wt. (g) (25 grams Max.)							
mL initial filtrate							
<small>(If miscible add back to final filtrate. If not, call initial filtrate Part A and final filtrate Part B of sample #)</small>							
mL of extraction fluid							
Speed of Rotary Device (rpm)							
TCLP/ZHE Completion							
Final pH of Leachate							
Date Completed							
Time Completed (18±2 hours)							
Filter Weighed							
Date Calibrated							
Analyst @ Completion							
Location							
Additional Notes							

*Note: A blank must be leached after every 20th sample has been extracted in each ZHE unit.

$$\text{Adjusted amount of sample to yield 25grams} = (25\text{grams}/\text{percent wet solids}) \times 100$$

Attachment 20.6
US Environmental Protection Agency Memorandum 35, Pg 10

US Environmental Protection Agency Memorandum #35, Page 10

Oily Waste Analysis

One of the most frequently asked questions on the MIC service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Leenik at (202) 260-7459. For additional information on microwave digestion contact Ollie Foran at (202) 260-4778.

The use of purge-and-trap (Method 8030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3010) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

Attachment 20.7
 TCLP 2-Phase Calculation Spreadsheet



Sample	Analyte	TCLP Limit (mg/L)	Phase 1 Result	Phase 1 RL	Phase 1 Units (mg/L or mg/kg)	Phase 1 Bulk Density (gm/mL)	Total Phase 1 Volume or Mass	Total Phase 1 Volume or Mass units	Phase 2 Result	Phase 2 RL	Phase 2 Units (mg/L or mg/kg)	Phase 2 Bulk Density (gm/mL)	Total Phase 2 Volume or Mass	Total Phase 2 Volume or Mass units	Combined TCLP Result (mg/L)	Combined TCLP RL (mg/L)
1205454-01	Benzene	0.5														
1205454-01	MEK	200			0	0	0	0			0	-	0	0		
1205454-01	Carbon Tetrachloride	0.5			0	0	0	0			0	-	0	0		
1205454-01	Chlorobenzene	100			0	0	0	0			0	-	0	0		
1205454-01	Chloroform	5			0	0	0	0			0	-	0	0		
1205454-01	1,2-Dichloroethane	0.5			0	0	0	0			0	-	0	0		
1205454-01	1,1-Dichloroethene	0.7			0	0	0	0			0	-	0	0		
1205454-01	Tetrachloroethene	0.7			0	0	0	0			0	-	0	0		
1205454-01	Trichloroethene	0.5			0	0	0	0			0	-	0	0		
1205454-01	Vinyl Chloride	0.2			0	0	0	0			0	-	0	0		
1205454-01	1,4-Dichlorobenzene	7.5														
1205454-01	2,4-Dinitrotoluene	0.13			0	0	0	0			0	0	0	0		
1205454-01	Hexachlorobenzene	0.13			0	0	0	0			0	0	0	0		
1205454-01	Hexachlorocyclopentadiene	0.5			0	0	0	0			0	0	0	0		
1205454-01	Hexachloroethane	3			0	0	0	0			0	0	0	0		
1205454-01	2-Methylphenol	200			0	0	0	0			0	0	0	0		
1205454-01	3-Methylphenol	200			0	0	0	0			0	0	0	0		
1205454-01	4-Methylphenol	200			0	0	0	0			0	0	0	0		
1205454-01	Nitrobenzene	2			0	0	0	0			0	0	0	0		
1205454-01	Pentachlorophenol	100			0	0	0	0			0	0	0	0		
1205454-01	Pyridine	5			0	0	0	0			0	0	0	0		
1205454-01	2,4,5-Trichlorophenol	400			0	0	0	0			0	0	0	0		
1205454-01	2,4,5-Trichlorophenol	2			0	0	0	0			0	0	0	0		
1205454-01	Arsenic	5	-0.1363	0.1	mg/kg	0.8725	348	mL	0.0065	0.5	mg/L	-	122.02	gm	-0.014	0.149
1205454-01	Barium	100	0.96473	0.1	mg/kg	0.8725	348	mL	0.01362	0.35	mg/L	-	122.02	gm	0.019	0.317
1205454-01	Cadmium	1	-0.0012	0.02	mg/kg	0.8725	348	mL	0.00081	0.05	mg/L	-	122.02	gm	0.001	0.046
1205454-01	Chromium	5	0.1919	0.1	mg/kg	0.8725	348	mL	-0.0007	0.25	mg/L	-	122.02	gm	0.020	0.230
1205454-01	Copper	100			mg/kg	0.8725	348	mL			mg/L	-	122.02	gm		
1205454-01	Lead	5	-1E-04	0.1	mg/kg	0.8725	348	mL	0.00363	0.25	mg/L	-	122.02	gm	0.008	0.230
1205454-01	Mercury	0.2		0.05	mg/kg	0.8725	348	mL	0.0002	0.0002	mg/L	-	122.02	gm		0.005
1205454-01	Selenium	1	-0.0021	0.1	mg/kg	0.8725	348	mL	0.01757	0.2	mg/L	-	122.02	gm	0.015	0.186
1205454-01	Silver	5	0.02662	0.02	mg/kg	0.8725	348	mL	-0.0015	0.05	mg/L	-	122.02	gm	0.002	0.046
1205454-01	Zinc	500			mg/kg	0.8725	348	mL			mg/L	-	122.02	gm		
1205454-01	2,4-D	10			0	0	0	0			0	0	0	0		
1205454-01	Silver	1			0	0	0	0			0	0	0	0		
1205454-01	Chordane	0.03			0	0	0	0			0	0	0	0		
1205454-01	Ercorn	0.02			0	0	0	0			0	0	0	0		
1205454-01	Heptachlor	0.008			0	0	0	0			0	0	0	0		
1205454-01	Heptachlor Epoxide	0.005			0	0	0	0			0	0	0	0		
1205454-01	Lindane	1			0	0	0	0			0	0	0	0		
1205454-01	Toxaphene	5			0	0	0	0			0	0	0	0		



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: 9-9-11
Denise S. Coffey

QA Officer: Date: 9-9-11
Tom C. Boucher

President: Date: 9-9-2011
Douglas Kriscunas

Procedure Number: GR-01-123
Revision Number: 5.8

Date Initiated: 5/6/9
Effective Date: 9/25/11

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By: David W. Johnson
Total Number of Pages: 21

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If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
<u>10-04-12</u>	<u></u>	<u>10-04-13</u>
_____	_____	_____
_____	_____	_____

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 0.05 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 II, 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 II, 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 Hg²⁺ state. All Hg²⁺ with an HCl carrier enters a gas-liquid separator where it is mixed with stannous chloride to form elemental mercury vapor (Hg⁰) according to the following equation:



- 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/Microplate 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/Salahad 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 Hg⁰ and Hg²⁺ 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 0 - 6° 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 μ L

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. The software version may be updated without notice if it performs at least as well as the older version.

9.10.1 Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

9.10.2 Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

9.11 Filter, 0.45 μ m 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 contract 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 Laboratory Reagent Water from the MilliQ System.
- 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 PROCEDURES

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 continuing calibration verification (CCV). The CCV is prepared at 2.0 µg/L and 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 CCV 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.

15.2.2.4 Remake and redigest the calibration standards if necessary.

- 15.2.2.5 Remake the CCV standard if necessary.
- 15.2.3 After resolving the problem, recalibrate the instrument and run the CCV successfully.
- 15.2.4 Digestions may not be analyzed without an acceptable CCV.
- 15.3 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 90 – 110% .
- 15.3.1 If the SCV fails recovery, repeat the analysis.
- 15.3.2 If the second analysis fails, initiate corrective action:
- 15.3.2.1 Check to make sure the instrument is operating correctly.
- 15.3.2.2 Recalibrate.
- 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.
- 15.4 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: Make all digestates before analysis. Solid matrix digestates must be filtered through a 0.45 μ m filter before analysis.

15.9 Check all raw data to make sure each result is within the calibration range. If any result is 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 Collect data from the instrument and transfer to the laboratory information management system in accordance with TriMatrix SOP GR-10-123.

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, handling 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 "command" 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) and one 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 laboratory reagent 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 with a known concentration of mercury.

18.3.2.1 Calculate BS recovery as follows:

$$\text{Percent Recovery} = \frac{C_{\text{Spike}}}{\text{Spike Quantity}} \times 100$$

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 BS must be within 85 – 115% of the expected value for method reference 245.1 and within 80 – 120% for method reference 7470A and 7471A.

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:

$$\text{Percent Recovery} = \frac{C_{\text{Spike}} - C_{\text{Orig}}}{\text{Spike Quantity}} \times 100$$

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:

$$\text{Relative Percent Difference} = \frac{\left| \frac{C_{MSD} - C_{MS}}{C_{MSD} + C_{MS}} \right|}{2} \times 100$$

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 80 – 120% of the expected recovery for method reference 7470A/ 7471A and 70 – 130% for method reference 245.1. MSD relative percent difference must be $\leq 20\%$.

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 AND when the leachate concentration is within 20% of the mercury regulatory limit (0.16 – 0.20 mg/L) but not over it.

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 70% 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 85 – 115% recovery for method reference 245.1 and 80 – 120% for method reference 7470A/7471A. Standard deviation of the average must be $\leq 20\%$ 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-01-123 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 II, 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 II, 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



PSA Maintenance Logbook

Date	Analyst	Emission/Reference Readings	Changed Pump Tube	Changed Lamp	Changed Probe	Replaced Sample Valve	Comments
5/6/93	DM						Replaced distal tube
5/13/93	NSS	116.4 104.5	✓				
5/18/93	NSS	200.1 116.4	✓				
5/20/93	NSS	185.9 114.1	✓				
5/26/93	NSS	190.1 112.1	✓				
5/27/93	NSS	195.1 114.1	✓				
5/27/93	NSS	190.4 112.7	✓				
5/28/93	NSS	193.1 113.1	✓				
5/28/93	NSS	193.9 114.3	✓				replaced GLS line & Drive Tube
5/28/93	NSS	200.1 113.2	✓				
5/28/93	NSS	200.0 112.7	✓				
5/28/93	NSS	180.9 107.9	✓				
5/28/93	NSS	194.5 117.1	✓				
5/28/93	NSS	184.1 110.9	✓				replaced GLS line & Drive Tube
5/28/93	NSS	190.3 107.9	✓				
5/28/93	NSS	175.1 114.0	✓				
5/28/93	NSS	200.1 115.0	✓				
5/28/93	NSS	200.2 114.8	✓				
5/28/93	NSS	200.5 115.1	✓				



SOP Name: Mercury by semi-Automated Cold-Vapor Atomic Absorption
EPA Method 245.1, SW-846 Method 7470A, SW-846 Method 7471A
SOP Number: GR-01-123 page 18 of 21

Revision Number: 5.8
Date Revised: 9/9/11
Date Initiated: 5/6/93

Attachment 23.2
Sample Identification and Weighing Sheet Example – Aqueous



Metals Laboratory Instrument Run Sequence Hg Aqueous

Instrument #: 216
Date: _____
Analyst: _____
SnCl #: 8110207

Data File: _____
Calibration BLK/ICB/CCB: 0.5% HNO3 LSP #1108080
Std/SCV prep date/analyst: _____

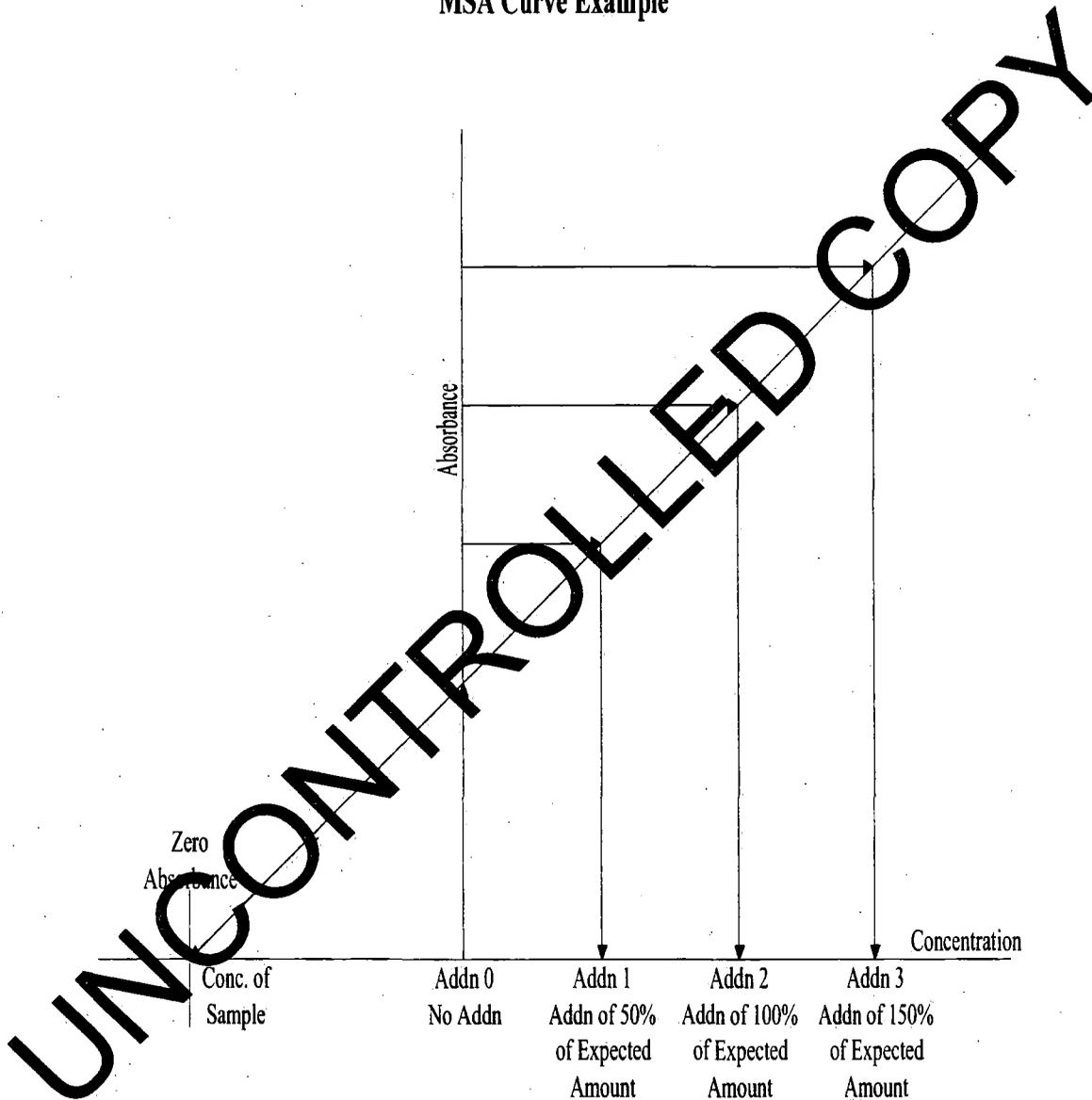
Standards/SCV prepped daily
Autosampler Position:

- 1. BLK/ICB/CCB/8050775
- 2. STD1/CRU/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 8109311

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				

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Attachment 23.3
MSA Curve Example



SOP Name: Mercury by semi-Automated Cold-Vapor Atomic Absorption
 EPA Method 243.1, SW-846 Method 7470A, SW-846 Method 7471A
 SOP Number: GR-01-123 Page 20 of 21

Revision Number: 5.8
 Date Revised: 9/9/11
 Date Initiated: 5/6/93

Attachment 23.4
Internal Chain-of-Custody Example

14-MAY-2003

TRIMATRIX LABORATORIES, INC.
 CHAIN OF CUSTODY REPORT FOR AN ENTIRE SUBMITTAL

PAGE 16

CLIENT: IDEQ-RSD
 SUBMITTAL: May 7 & 8, 2003 Soils

PROJECT: Former Osceola Refinery
 West Branch-Sag. Bay Dist.
 PROJECT: 337-7

Parameter: MERCURY, TOTAL

Method: CV/AS 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	<i>m. a. scott</i>			<i>05-05-03 3:14 pm</i>	<i>05-15-03 3:14 pm</i>
331323	<i>MSS</i>				
331324	<i>MSS</i>				
331325	<i>MSS</i>				
331326	<i>MSS</i>				
331327	<i>MSS</i>				
331328	<i>MSS</i>				
331329	<i>MSS</i>				
331330	<i>MSS</i>				
331331	<i>MSS</i>				
331332	<i>MSS</i>				
331333	<i>MSS</i>				
331334	<i>MSS</i>				
331335	<i>MSS</i>				
331336	<i>MSS</i>				
331337	<i>MSS</i>			<i>05-13-03 1:30 pm</i>	<i>05-15-03 3:30 pm</i>

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SOP Name: Mercury by semi-Automated Cold-Vapor Atomic Absorption
EPA Method 245.1, SW-846 Method 7470A, SW-846 Method 7471A
SOP Number: GR-01-123 page 21 of 21

Revision Number: 5.8
Date Revised: 9/9/11
Date Initiated: 5/6/93

Attachment 23.5
Sample Identification and Weighing Sheet Example – Solid



Metals Laboratory Instrument Run Sequence Hg Solids

Instrument #: 216 Date: _____ Data File: _____
Date: _____ Calibration BLK/ICB/CCB: 0.5% HNO₃ LOT# 108080
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
- 5. 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									
17					51				
18					52				
19					53				
20					54				
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31					65				
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33					67				
34					68				
35					69				
36					70				
37					71				
38					72				
39					73				
40					74				
41					75				
42					76				
43					77				
44					78				



STANDARD OPERATING PROCEDURE

Block Digestion of Solids for ICP and ICPMS

SW-846 Method 3050B

APPROVALS:

Area Supervisor: _____

Marge A. Scott
Marge A. Scott

Date: _____

10-18-12

QA Officer: _____

Tom Bocher
Tom Bocher

Date: _____

10-22-12

Laboratory President: _____

Douglas E. Kriscunas
Douglas E. Kriscunas

Date: _____

10-19-2012

Procedure Number: GR-01-137

Revision Number: 1.7

Date Initiated: 2/05/01

Effective Date: 10/31/12

Date Revised: 10/18/12

Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 17

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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 to 0.55 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		ICP or ICPMS			
Aluminum	Silicon	Antimony	Chromium	Nickel	Zinc
Calcium	Sodium	Arsenic	Cobalt	Selenium	Strontium
Iron	Strontium	Barium	Copper	Silver	
Lithium	Titanium	Beryllium	Lead	Thallium	
Magnesium		Boron	Manganese	Tin	
Potassium		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-09-128, *Homogenization, Grinding and Drying of Solid Samples*, 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
- 5.9 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, 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 Wear a laboratory coat and approved safety glasses while in the digestions 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.
- 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 exposure 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 Waste samples can be highly toxic and corrosive. Treat any exposure as a potential danger and immediately decontaminate the exposed surface. Clean waste-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 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-09-178. 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 μ L
- 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° F 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 (HNO₃), concentrated, trace metal grade
- 11.3 Prepare 1% (v/v) HNO₃ as follows:
- 11.3.1 Carefully measure 50 mL of HNO₃ to 50 mL of laboratory reagent water.
- 11.3.2 Prepare fresh daily.
- 11.4 Prepare 5% (v/v) HNO₃ as follows:
- 11.4.1 Carefully measure 5 mL of HNO₃ 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 (H₂O₂), 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 to 0.55 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 1.0 (v/v) HNO₃. 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 HNO₃ 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% H₂O₂ 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% H₂O₂. 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 $95^\circ \pm 5^\circ \text{C}$ 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 on 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 and gold 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 $95^\circ \pm 5^\circ \text{C}$ 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 – 0.55 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 $95^\circ \pm 5^\circ \text{C}$ 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 ($\sim 95^\circ \text{C}$) 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 ($\sim 95^\circ \text{C}$) concentrated HCl. Return to the block digester with a non-ribbed watchglass and heat at $95^\circ \pm 5^\circ \text{C}$ 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 to volume, digestates are ready for analysis by ICP (tin) or ICPMS (antimony).

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 TriMatrix 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) HNO₃ into a digestion cup and spike with 250 µL 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) HNO₃ into a digestion cup and spike with 20 µL 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 – 0.55 g of dried sample into a digestion cup and spike with 250 µL 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 – 0.55 g of dried sample into a digestion cup and spike with 20 µL 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 begins to boil 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 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 A Method Detection Limit (MDL) study must be performed in accordance with TriMatrix SOP GR-10-125, including quarterly verifications.

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-SSG-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**



CERTIFICATE OF ANALYSIS

tel: 800.669.6777 - 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 Principles."

2.0 DESCRIPTION OF CRM Custom Solution
 Catalog No.: TRIMATRIX-SSW-1A
 Lot Number: Y-MEB168079
 Matrix: 2% HNO3(abs)

2,500.00 µg/mL each: Ca, K, Mg, Na.
 250.00 µg/mL each: Al, As, P, Se, Ti.
 50.00 µg/mL each: Ag, B, Ba, Cd, Cr, 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	249.5 ± 0.7 µg/mL	Arsenic, As	250.2 ± 0.8 µg/mL	Barium, Ba	50.16 ± 0.08 µg/mL
Beryllium, Be	5.00 ± 0.10 µg/mL	Boron, B	50.07 ± 0.10 µg/mL	Cadmium, Cd	50.16 ± 0.22 µg/mL
Calcium, Ca	2,500 ± 8 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	9.91 ± 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	249.5 ± 0.8 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	50.06 ± 0.11 µg/mL	Thallium, Tl	249.9 ± 0.7 µg/mL	Vanadium, V	49.98 ± 0.14 µg/mL
	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:

$$\text{Certified Value } (\bar{x}) = \frac{\sum x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2(\sum s_i^2)^{1/2}}{(n)^{1/2}}$$

(\bar{x}) = mean
 x_i = individual results
 n = number of measurements
 $\sum s_i$ = 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.)

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A607108
DA
7-06-06

SOP Name: Block Digestion of Solids for ICP and ICPMS
SW-846 Method 3050B

Revision Number: 1.7
Date Revised: 10/18/12
Date Initiated: 2/6/01

SOP Number: GR-01-137

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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

CERTIFICATE OF ANALYSIS

tel: 800.669.6777 / 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 Principles."

2.0 DESCRIPTION OF CRM Custom Solution
Catalog No.: TRIMATRIX-SSW-SSS-2A
Lot Number: Y-MEB201134
Matrix: tr. HF, 2% HNO₃ (v/v)

A609280
ORL 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	250.8 ± 0.8 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

$$\text{Certified Value } (\bar{x}) = \frac{\sum x_i}{n}$$

(\bar{x}) = mean
 x_i = individual results
 n = number of measurements

$$\text{Uncertainty } (u) = \frac{2(\sum \epsilon_i)^2}{n}^{1/2}$$

$\sum \epsilon_i$ = 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 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

<u>Source</u>	<u>Source Purity</u>	<u>Matrix</u>	<u>Standard Concentration</u>
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 acids have 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

Exp Date:
 MSDS **ATTACHED**

400 Wando Park Blvd.
 Mt. Pleasant, South Carolina 29464
 Phone: 803.881.6560
 Toll Free: 1.800.343.5319
 FAX: 1.843.881.3964
 www.environmentalexpress.com

*AL602735
 et p 3-31-07
 Paul 2-28-06*



Attachment 20.4
ICP/MS Spiking Solution B Certificate of Analysis Example

ENVIRONMENTAL EXPRESS

Certificate of Analysis

HP2079-1-1L
Solution B
Lot # 605319

<u>Source</u>	<u>Source Purity</u>	<u>Matrix</u>	<u>Standard Concentration</u>
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. 3109 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

Exp Date: MSS ATTACHED

490 Wanda 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



*A602736
exp 2-21-07
red 2-28-06*



SOP Name: Block Digestion of Solids for ICP and ICPMS
 SW-846 Method 3050B
 SOP Number: GR-01-137

Revision Number: 1.7
 Date Revised: 10/18/12
 Date Initiated: 2/6/01

**Attachment 20.5
 Work Order Report Example**

TriMatrix Laboratories, Inc.

WORK ORDER 0609330

Printed: 7/11/2007 5:33:43PM

Page 1 of 6

Client: [REDACTED]	Project Manager: Gary L. Wood
Project: [REDACTED]	Project Number: [none]
Work Order: [REDACTED]	SDG: [REDACTED]

Report To:

[REDACTED]
 [REDACTED]
 [REDACTED]
 [REDACTED]

Invoice To:

[REDACTED]
 [REDACTED]
 [REDACTED]

Client Due Date: Oct-04-06 16:00 (10 day TAT)
 Date Received: Sep-20-06 12:00 25.9°C
 Date Logged In: Sep-21-06 16:37
 W.O. Comments: 3FL

Report Level: SFL
 Received By: William J. Cole
 Logged In By: Kristina N. Courtney

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

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SOP Name: Block Digestion of Solids for ICP and ICPMS
SW-846 Method 3050B

Revision Number: 1.7
Date Revised: 10/18/12
Date Initiated: 2/6/01

SOP Number: GR-01-137

page 17 of 17

Attachment 20.6
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0610939 Page 1 of 3

Printed: 7/11/2006 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	Se 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 OC 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	A607108	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	A607108	250	MATRIX SPIKE DUP	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50						
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50						
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50						
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50						
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50						
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50						
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50						
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50						
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50						
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50						
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50						
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50						
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50						
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50						
0609330-03	A	Sep-25-06 07:00	MAS	0.4995	50						
0609330-03	A	Sep-25-06 07:00	MAS	0.4995	50						

Comments:	Analyst Initials:
-----------	-------------------

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STANDARD OPERATING PROCEDURE

Block Digestion of Total Metals in Water for ICP Method 6040C

SW-846 Method 3010A

APPROVALS:

Area Supervisor: Marge A. Scott
Marge A. Scott

Date: 09/05/12

QA Officer: Tom C. Booher
Tom C. Booher

Date: 9-7-12

Laboratory President: Douglas E. Krisunas
Douglas E. Krisunas

Date: 9/7/2012

Procedure Number: GR-01-147
Revision Number: 0.5

Date Initiated: 7/15/03
Effective Date: 9/15/12

Date Revised: 8/30/12
Pages Revised: All

By: Marge A. Scott
Total Number of Pages: 15

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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 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 I, 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.

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-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision
- 5.4 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, 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.

7.0 SAFETY PRECAUTIONS

- 7.1 Wear a laboratory coat and approved safety glasses while in the 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.
- 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 exposure 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 waste-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 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.

8.2.1 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.

8.2.2 If the sample must be analyzed within 24 hours of collection, the sample must be preserved immediately during collection.

8.2.3 If boron is an analyte of interest the sample must be preserved immediately during collection.

8.2.4 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 μ L
- 9.2 Block digester 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 digester 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 (HNO₃), 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 digester. 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 digester 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 HNO₃ 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 HNO₃. 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 HNO₃ then continue refluxing as before. Repeat 0.75 mL HNO₃ 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 (refer to Attachment 20.3).
- 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 TriMatrix Quality Assurance 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.
- 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 200 µL of solution SSW-1A and 200 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 matrix spike and matrix spike duplicates by measuring a 25.0 mL sample aliquot into a digestion cup, then spiking with 200 µL of solution SSW-1A and 200 µL of solution SSW/SSS-2A.

15.6 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.

157 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 and approved. Give copies of all 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 then submit to QA.

16.2 A Method Detection Limit (MDL) Study is required in accordance with SOP GR-10-125 for 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 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 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 I, Revision 1, July 1992, Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy"*

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

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Attachment 20.1
ICP Spiking Solution SSW-1A Certificate of Analysis Example



195 Lehigh Avenue, Suite 4
 Lokewood, New Jersey 08701 · USA
 info@inorganicventures.com

CERTIFICATE OF ANALYSIS

tel: 800.669.6777 / 2.901.1900
 fax: 2.901.1903
 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 Principles."

2.0 DESCRIPTION OF CRM Custom Solution
 Catalog No.: TRIMATRIX-SSW-1A
 Lot Number: Y-MEB188079
 Matrix: 2% HNO3(abs)

*AG07108
 DA
 7-06-06*

2,500.00 µg/mL each:
 Ca, K, Mg, Na,
 250.00 µg/mL each:
 Al, As, P, Se, Ti,
 50.00 µg/mL each:
 Ag, B, Ba, Cd, Co, Cr, 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	60.16 ± 0.08 µg/mL
Beryllium, Be	60.04 ± 0.05 µg/mL	Boron, B	60.07 ± 0.10 µg/mL	Cadmium, Cd	60.16 ± 0.22 µg/mL
Calcium, Ca	250.0 ± 0.6 µg/mL	Chromium+3, Cr3	60.11 ± 0.16 µg/mL	Cobalt, Co	60.09 ± 0.12 µg/mL
Copper, Cu	60.17 ± 0.14 µg/mL	Iron, Fe	49.84 ± 0.32 µg/mL	Lead, Pb	60.17 ± 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	60.10 ± 0.12 µg/mL	Phosphorus, P	249.5 ± 0.6 µg/mL	Potassium, K	2,500 ± 5 µg/mL
Selenium, Se	249.8 ± 0.6 µg/mL	Silver, Ag	60.01 ± 0.11 µg/mL	Sodium, Na	2,497 ± 1 µg/mL
Strontium, Sr	60.06 ± 0.11 µg/mL	Thallium, Tl	249.9 ± 0.7 µg/mL	Vanadium, V	49.88 ± 0.14 µg/mL
Zinc, Zn	60.09 ± 0.12 µg/mL				

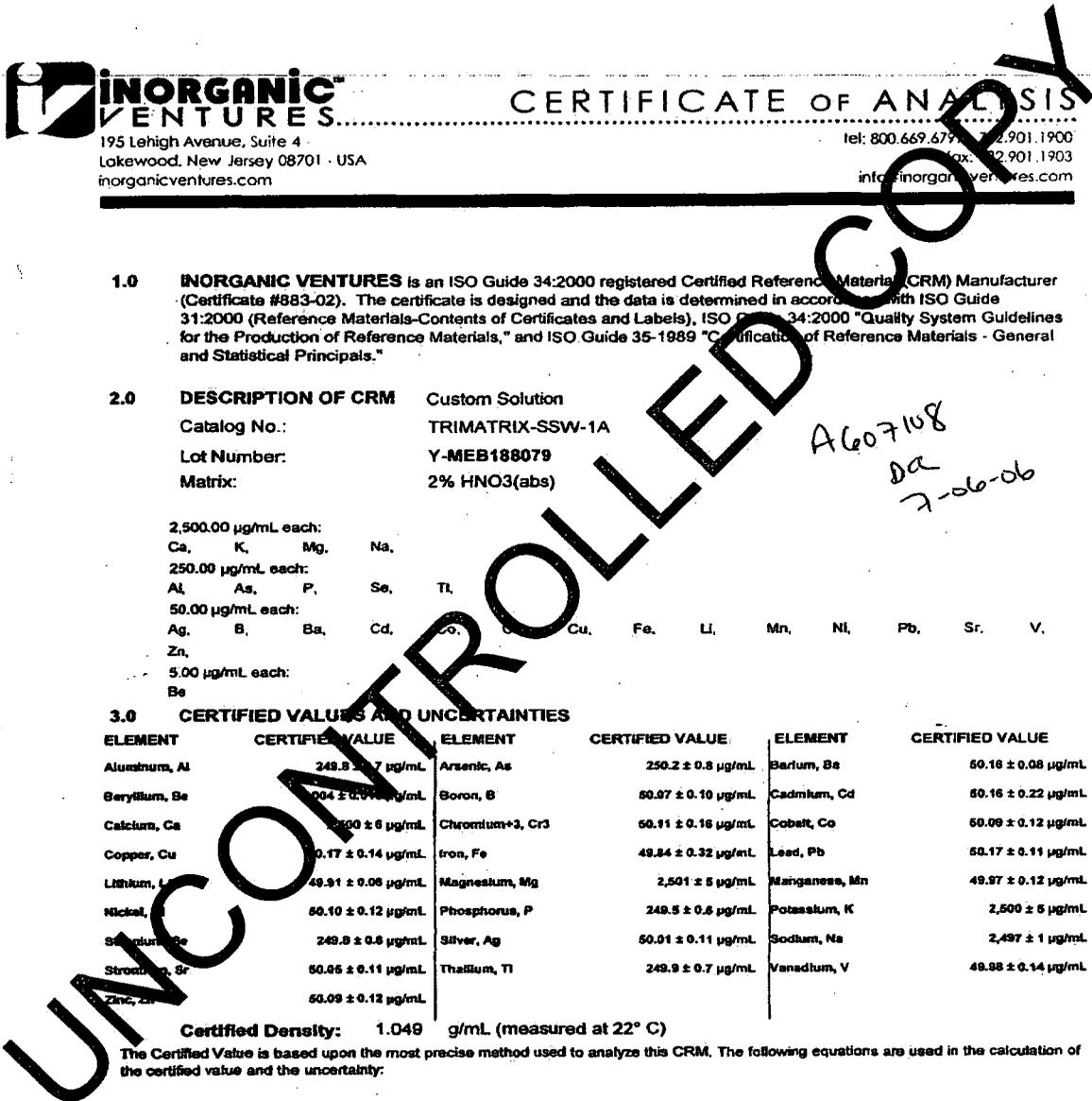
Certified Density: 1.049 g/mL (measured at 22° C)

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:

$$\text{Certified Value } (\bar{x}) = \frac{\sum x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\sum \epsilon_i)^2]^{1/2}}{(n)}$$

(\bar{x}) = mean
 x_i = individual results
 n = number of measurements
 $\sum \epsilon_i$ = 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



CERTIFICATE OF ANALYSIS

tel: 800.646.6799 • 32.901.1900
fax: 32.901.1903
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 Principles."

2.0 DESCRIPTION OF CRM Custom Solution
Catalog No.: TRIMATRIX-SSW-SSS-2A
Lot Number: Y-MEB201134
Matrix: tr. HF, 2% HNO₃ (abs)

*A609280
OK 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	250.8 ± 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 on the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty.

$$\text{Certified Value } (\bar{x}) = \frac{\sum x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2((\sum s_i)^2)^{1/2}}{(n)^{1/2}}$$

(\bar{x}) = mean
 x_i = individual results
 n = number of measurements
 $\sum s_i$ = 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 8.10)

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**



WORK ORDER 1208168

Printed: 8/15/2012, 12:28:08PM

Page 1 of 2

Metals Sample Receipt Notice

Client: XXXXXXXXXX	Project Manager: Jennifer L. Rice
Project: Groundwater Testing	Project Number: [none]
Client Due Date: Aug-22-12 23:00 (10 day TAT)	Report Level: 2RLM
W.O. Comments: QC is 2RLM	

Lab Number	Sample Name Analysis	Matrix	Sampled Date TAT	Expires Date	Sample Comments	Lab Due Date	Comments
1208168-01	Monitoring Well #1	Water	Aug-03-12 14:45	Expires to meet			
	As Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	
	B Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Ba Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Ca Total 6010B		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Cd Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Cr Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Fe Total 6010B		10	Jan-30-13 14:45		Aug-21-12 17:00	
	K Total 6010B		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Li Total 6010B		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Mg Total 6010B		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Mn Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Mo Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Na Total 6010B		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Pb Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Sb Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	
	Se Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	
	V Total 6020		10	Jan-30-13 14:45		Aug-21-12 17:00	

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SOP Name: Block Digestion of Total Metals in Water for ICP Method 6010C
 SW-846 Method 3010A
 SOP Number: GR-01-147

Revision Number: 0.5
 Date Revised: 8/30/12
 Date Initiated: 3/17/03

Attachment 20.4
Preparation Batch Report Example

Trimatrix Laboratories, Inc.

PREPARATION BATCH **1209753** Page 1 of 2

Printed: 8/29/2012 4:41:36PM

Metals, Water, 3010A Digestion
 (No Surrogate)

Batch Comments: mas 08/10/12 7:00am-4:00pm

Standard	Description	Solvent	Lot/Num
1100114	TRIMATRIX-SSW-SSS-2A	3% HNO3 tr. HF	E2-MEB376075
1100115	TRIMATRIX-SSW-1A	3% HNO3	E2-MEB376074
2041322	TRIMATRIX-SSW-1A	3% HNO3	F2-MEB419106
2041323	TRIMATRIX-SSW-SSS-2A	3% HNO3 tr. HF	F2-MEB419105
2060256	1:1 HCL	51122	51122
2061357	Nitric Acid - lot 51132	N/A	51132

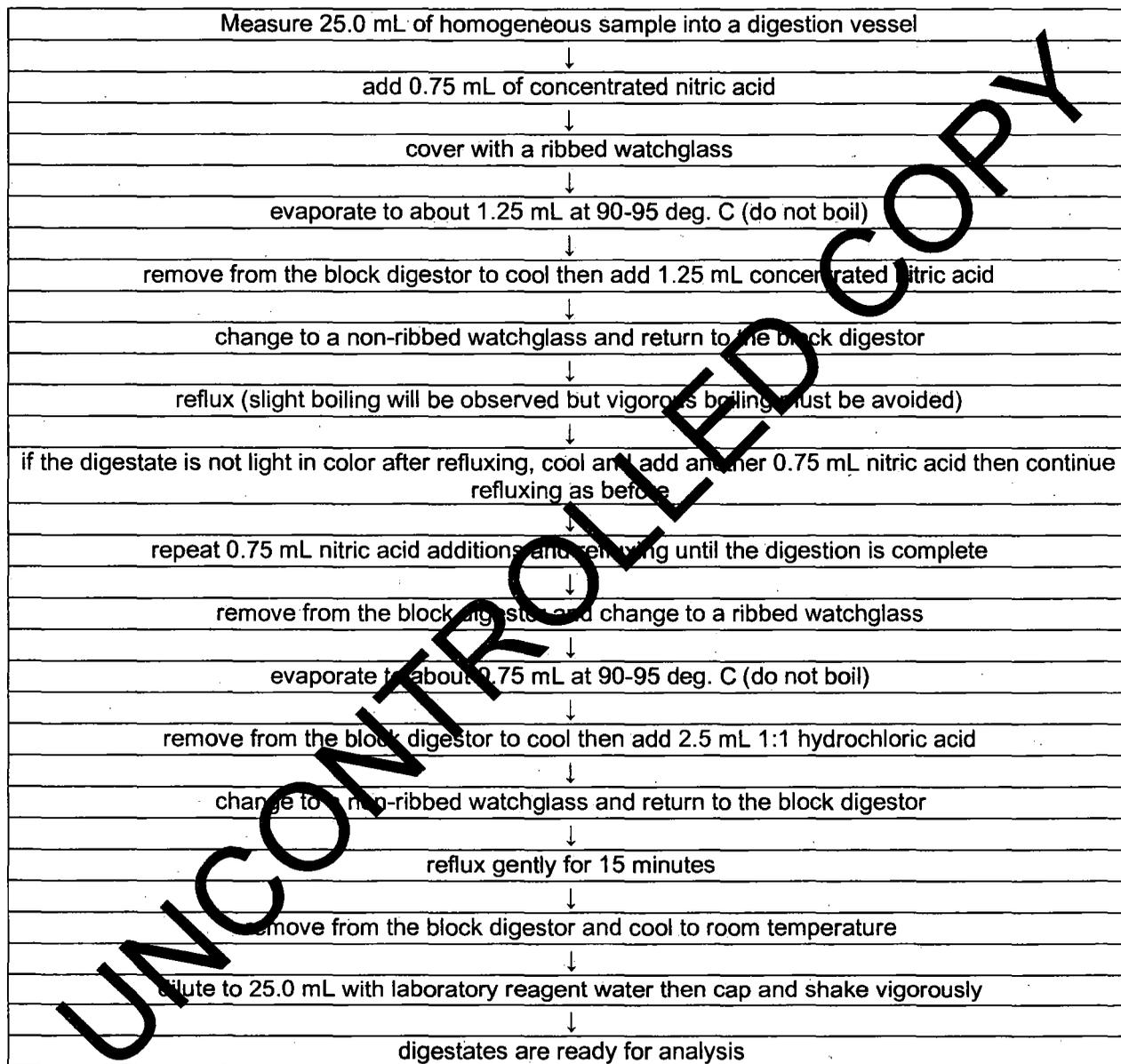
Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
1208168	Na Total 6010B	1208168	Mn Total 6010B	1208168	Mg Total 6010B
1208168	Li Total 6010B	1208168	K Total 6010B	1208168	Fe Total 6010B
1208168	Ca Total 6010B	1208171	Fe Total 6010B	1208173	Fe Total 6010B

Balance ID: _____ pH Meter: _____

Lab Number	Container	Prepared	By	Initial (ml)	Final (ml)	Surrogate	Source ID	Spike ID	Volume	Notes / Use	Additional Comments
1209753-ELK1		Aug-10-12 07:00	MAS	25	25				200	BLANK	
1209753-EG1		Aug-10-12 07:00	MAS	25	25			2041322	200	GC	
1209753-MS1		Aug-10-12 07:00	MAS	25	25		1208168-C1	2041322	200	MATRIX SPIKE	
1209753-MSD1		Aug-10-12 07:00	MAS	25	25		1208168-C1	2041322	200	MATRIX SPIKE DLP	
1208168-01	A	Aug-10-12 07:00	MAS	25	25						
1208168-01	A	Aug-10-12 07:00	MAS	25	25						
1208168-01	A	Aug-10-12 07:00	MAS	25	25						
1208168-01	A	Aug-10-12 07:00	MAS	25	25						
1208168-01	A	Aug-10-12 07:00	MAS	25	25						Added for SequenceQC in: 2116034
1208168-01	A	Aug-10-12 07:00	MAS	25	25						
1208168-01	A	Aug-10-12 07:00	MAS	25	25						
1208171-01	B	Aug-10-12 07:00	MAS	25	25						
1208171-02	B	Aug-10-12 07:00	MAS	25	25						
1208171-03	B	Aug-10-12 07:00	MAS	25	25						

Comments: _____ Analyte Initials: _____

bci_TaMatrix.rpt

Attachment 20.5
Digestion Flowchart




STANDARD OPERATING PROCEDURE

Determination of Inorganic Anions by Ion Chromatography

EPA Method 300.0
SW-846 Method 9056A

APPROVALS:

Area Supervisor: Heather L. Brady Date: 8-4-12
Heather L. Brady

QA Officer: Tom E. Leach Date: 8-8-12
Tom E. Leach

Laboratory President: Douglas E. Kriscunas Date: 8/6/2012
Douglas E. Kriscunas

Procedure Number: GR-02-113
Revision Number: 3.0

Date Initiated: 4/1/00
Effective Date: 8/20/12

Date Revised: 8/3/12
Pages Revised: All

By: Katie A. Root
Total Number of Pages: 21

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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 determination of inorganic anions in a variety of matrices including drinking water, surface water, mixed domestic and industrial wastewater, groundwater, laboratory reagent waters, solids (using the extraction procedure given in Section 13.2), and leachates not containing acetic acid.
- 1.2 The analysis range tested for each anion is as follows:

Analyte	Range, (mg/L)
Bromide, Fluoride, Nitrite-N, Nitrate-N, o-Phosphate-P	0.05 to 5
Chloride	1 to 100
Sulfate	1 to 100
Iodide	0.005 to 0.100

Note: The calibration ranges above are based on a 25 µL sample loop with the exception of iodide, which is based on a 1000 µL sample loop.

- 1.3 This procedure is recommended for use only by analysts experienced in the use of ion chromatography and in the interpretation of ion chromatograms.
- 1.4 Bromide and nitrite react with most oxidants employed as disinfectants in potable water. The feasibility of measuring anions in treated water must be considered prior to conducting the analysis.

2.0 PRINCIPLE METHOD REFERENCES

- 2.1 EMSL, Office of Research and Development, "Determination of Inorganic Anions by Ion Chromatography". USEPA Method 300.0; USEPA, Cincinnati, OH 45268, USA, rev. 2.1, 1993
- 2.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Revision 1 February 2007, Method 9056A, "Determination of Inorganic Anions by Ion Chromatography"

3.0 SUMMARY OF PROCEDURE

- 3.1 A small sample volume is introduced into the ion chromatograph. Anions of interest are separated and measured using a guard column, analytical column, suppressor cartridge and conductivity detector.
- 3.2 The dissolution procedure described in Section 13.2 must first be performed when using this procedure for solids. Results from solid samples must be reported as "soluble", not "total".

4.0 PARAMETER OR COMPOUND LIST

- 4.1 This procedure covers determination of the following inorganic anions:

Bromide	Nitrite Nitrogen
Chloride	ortho-Phosphate as Phosphorus
Fluoride	Sulfate
Nitrate Nitrogen	Iodide

5.0 REFERENCED SOPs

- 5.1 TriMatrix Laboratories SOP GR-15-102, *Laboratory Waste Disposal*, latest revision
- 5.2 TriMatrix Laboratories SOP GR-16-103, *Glassware Cleaning and Preparation for the Wet Chemistry and Metals Laboratory*, latest revision
- 5.3 TriMatrix Laboratories SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision
- 5.4 TriMatrix Laboratories SOP GR-10-115, *Manual Integrations*, latest revision
- 5.5 TriMatrix Laboratories SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision
- 5.6 TriMatrix Laboratories SOP GR-16-117, *Extraction of Soluble Inorganic Analytes From Soil*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

- 6.1 Interferences can be caused by substances with a retention time similar to but overlapping the analyte of interest. Also, large analyte concentrations in a sample can interfere with the peak resolution of a closely eluting analyte. Sample dilution and/or fortification can be used to solve many interference problems associated with overlapping retention times. However, a sample dilution will proportionally elevate reporting limits.
- 6.2 Method interferences are caused by contaminants in laboratory reagent water, reagents, glassware and other sample processing apparatus. These may lead to discrete artifacts or an elevated baseline in ion chromatograms.
- 6.3 Any anion, eluted or un-retained by the column will elute in the area of fluoride and interfere. Known co-elution is caused by simple organic anions. At concentrations of fluoride above 1.5 mg/L this interference may not be significant. Unretained peaks such as low molecular weight organic acids (formate, acetate, propionate) which are conductive and co-elute with or near fluoride and bias fluoride quantitation must be avoided.
- 6.4 The acetate anion elutes early during the chromatographic run. Targeted anion retention times shift when a large concentration of acetate is present. Therefore, this procedure is not used for leachates of samples when acetic acid is used for pH adjustment.

7.0 SAFETY PRECAUTIONS

- 7.1 Wear a laboratory coat and approved safety glasses while in the laboratory. In addition, disposable gloves must be worn whenever samples and/or reagents are handled.

7.2 Follow all instructions outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan to minimize injuries and accidents.

7.3 For laboratory waste disposal, refer to TriMatrix SOP GR-15-102.

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 exposure 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 Waste samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste contaminated personal protective equipment before using again.

7.6 Bring all safety issues to the immediate 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 plastic or glass bottles. All bottles must be certified clean. Volumes collected must be sufficient to ensure a representative sample, allow for replicate analysis (if required) and minimize waste disposal.

8.2 Sample preservation and hold times are determined by the anion(s) of interest. In a given sample, the anion that requires the most preservation and shortest hold time will determine the preservation treatment, except where a chemical preservative addition will interfere with the analysis of other anion(s). Preservation and hold times for anions that can be determined by this procedure are as follows:

<u>Anion</u>	<u>Preservation</u>	<u>Holding Time</u>
Bromide	None required	28 days
Chloride	None required	28 days
Fluoride	None required	28 days
Nitrate Nitrogen	cool to 0 - 6° C	48 hours
Nitrite Nitrogen	cool to 0 - 6° C	48 hours
ortho-Phosphate as P	cool to 0 - 6° C	48 hours
Sulfate	cool to 0 - 6° C	28 days
Iodide	none required	28 days

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

- 9.1 Balance – analytical, capable of accurately weighing to the nearest 0.0001 g
- 9.2 Glassware – Class A volumetric flasks and autopipettes as required
- 9.3 Ion Chromatograph – Dionex ICS-2000 analytical system complete with ion chromatograph and all required accessories including analytical columns and detectors.
- 9.3.1 Anion guard column: Dionex AG-18, 2x50 mm, PN 060555 or Dionex AG-19, 2x50 mm, PN 062888
- 9.3.2 Anion profiling column: Dionex AS-18, 2x50 mm, PN 060553 or Dionex AS19, 2x250 mm, PN 062886
- 9.3.3 Anion suppressor cartridge: Dionex ASRS Ultra II, 2 mm, PN 061562
- 9.3.4 Detector: Dionex PN 057985
- 9.3.5 Autosampler: Dionex AS40 automated sampler, PN 0540-1
- 9.3.6 0.22 µm nylon filter
- 9.3.7 0.45 µm syringe filter
- 9.3.8 25 µL sample loop
- 9.3.9 1000 µL sample loop
- 10.0 ROUTINE PREVENTIVE MAINTENANCE AND TROUBLESHOOTING**
- 10.1 Keep the instrument clear of debris and promptly clean up spills.
- 10.2 For additional information on system maintenance and troubleshooting, refer to the Dionex operations manual and troubleshooting guide.
- 11.0 CHEMICALS AND REAGENTS**
- 11.1 Laboratory reagent water: Use ASTM Type II MilliQ system water containing particles no larger than 0.20 µm for all solutions.
- 11.2 Eluent, the mobile phase liquid that separates anions in the column is automatically generated by the instrument from laboratory reagent water and reagent potassium hydroxide.
- 12.0 STANDARDS PREPARATION**
- 12.1 Individual Stock Standard Solutions, 1000 mg/L (1 mg/mL)
- 12.1.1 Stock standards may be purchased as certified solutions or prepared from ACS reagent grade materials (dried at 105° C for 30 minutes) as listed below.

12.1.2 To separate 250 mL volumetric flasks, add the exact mass of reagent listed below and dilute to volume with laboratory reagent water. Stir to dissolve before dilution.

Stock Reagent	Exact Mass (gm)	Concentration (mg/L)
Sodium Fluoride (NaF)	0.5526	1000 mg F/L
Sodium Chloride (NaCl)	0.4121	1000 mg Cl/L
Sodium Nitrite (NaNO ₂)	1.2314	1000 mg NO ₂ ⁻ /L
Sodium Bromide (NaBr)	0.3219	1000 mg Br/L
Sodium Nitrate (NaNO ₃)	1.1170	1000 mg NO ₃ ⁻ /L
Potassium Phosphate (KH ₂ PO ₄)	1.0914	1000 mg HPO ₄ ⁻ -P/L
Potassium Sulfate (K ₂ SO ₄)	0.535	1000 mg SO ₄ ²⁻ /L
Potassium Iodide (KI)	0.2916 into 200 mL	1000 mg I ⁻ /L

12.1.3 Prepare a second set of individual stock solutions from sources dissimilar to those used above for use in the second-source calibration verification (SCV) standard. A separate lot of chemical from the same manufacturer or a different manufacturer may be used when preparing from neat materials.

12.3 Working Mixed Standard A

12.3.1 Transfer volumes of individual 1000 mg/L solutions (Section 12.1) into a single 200 mL volumetric flask as described below. Dilute to volume with laboratory reagent water and invert to mix. Store at 0 - 6° C.

Stock Standard	mL to add	Concentration (mg/L)
Fluoride, 1000 mg/L	1.0	5
Chloride, 1000 mg/L	20	100
Nitrite N, 1000 mg/L	1.0	5
Bromide, 1000 mg/L	1.0	5
Nitrate N, 1000 mg/L	1.0	5
0-Phosphate P, 1000 mg/L	1.0	5
Sulfate, 1000 mg/L	20	100
Iodide, 1000 mg/L (separately)	0.100 into 100 mL	1.0

12.3.2 Repeat the above preparation using the SCV individual stock solutions.

Note: The iodide SCV (0.04 mg/L) is prepared by measuring 4.0 mL of 1.0 mg/L I⁻ into 100 mL of laboratory reagent water in a volumetric flask.

12.4 Working Mixed Standards B through F

12.4.1 Working Mixed Standards B through F are prepared by diluting Working Mixed Standard A (Section 12.3) with laboratory reagent water as specified below. Standard A is repeated for reference.

Std	Standard A (mL)	Final Volume (mL)	Concentration (mg/L)						
			F ⁻	Cl ⁻	NO ₂ ⁻ N	Br ⁻	NO ₃ ⁻ N	HPO ₄ ²⁻ P	SO ₄ ²⁻
A	-	-	5.0	100	5.0	5.0	5.0	5.0	100.0
B	30.0	50	3.0	60	3.0	3.0	3.0	3.0	60.0
C	20.0	100	1.0	20	1.0	1.0	1.0	1.0	20.0
D	20.0	250	0.4	8.0	0.4	0.4	0.4	0.4	8.0
E	10.0	500	0.10	2.0	0.10	0.10	0.10	0.10	2.0
F	5.0	500	0.05	1.0	0.05	0.05	0.05	0.05	1.0

12.4.2 Example: To prepare standard B, add 30 mL of standard A to a 50 mL volumetric flask or bottle. Dilute to volume with laboratory reagent water to reach a total solution volume of 50 mL.

Iodide Standards Preparation, Separately				
Standards	Initial concentration (mg/L)	Initial volume (mL)	Final volume (mL)	Final Concentration (mg/L)
Standard A	1000	0.100	100	1.0
Standard 1	1.0	0.500	100	0.005
Standard 2	1.0	1.0	100	0.01
Standard 3	1.0	1.0	50	0.02
Standard 4	1.0	2.0	50	0.04
Standard 5	1.0	4.0	50	0.08
Standard 6	1.0	5.0	50	0.10

12.5 Individual stock solutions (Section 12.1) are stable for 1 month. Diluted stock solutions (Section 12.2) and mixed calibration standards (Sections 12.3 and 12.4) are prepared weekly except those that contain nitrite and o-phosphate which must be prepared fresh daily. Store at 0 - 6° C.

12.6 All standards must be uniquely identified and recorded in the standards log in the laboratory information management system (Element™).

13.0 SAMPLE PREPARATION

13.1 All samples, standards and quality control must be filtered either in-line as part of the analysis or externally using a 0.45 µm nylon filter to prevent damaging valves and columns.

13.2 Soil samples must be extracted prior to analysis in accordance with TriMatrix SOP GR-16-117.

13.2.1 Soil Analysis must be reported as "soluble" and not "total".

14.0 CALIBRATION PROCEDURES

14.1 A retention time study must be completed after any major changes to the IC system (ex. Changing the guard column or separation column).

14.1.1 Before establishing retention time windows, make sure the instrument is operating reliably and that system conditions are optimized to all targeted analytes.

14.1.2 After an initial calibration, use three injections of all target analytes over the course of a 24-hr period at the mid-point concentration of the calibration range.

Note: A convenient approach is to use the mid point standard of the initial calibration then two subsequent continuing calibration verification runs obtained throughout the course of the day.

14.1.3 Record the retention time for each analyte from the initial calibration and the three retention time study injections into the Retention Time Window Spreadsheet. See Attachment 23.1

14.1.4 The retention times of the first daily calibration verification standard is entered into the retention time window spreadsheet, in order for it to correctly calculate the retention windows for the day.

14.1.5 Whenever the observed retention time of a calibration verification standard is outside the established retention time window, determine the cause and correct the problem before continuing with sample analysis. If the observed retention time is greater than $\pm 10\%$ from the previous calibration or calibration verification, re-calibrate the instrument.

14.2 Analyte retention time is affected by eluent concentration, flow rate, analyte concentration and column performance. To adjust retention times, adjust the integration events table and peak table to ensure each peak is identified correctly and integrated properly. Resolution between peaks may be optimized as follows.

15.10.1 The eluent may be diluted 10 - 30 percent. Dilution will increase separation but will also cause later-eluting analytes to be retained longer.

15.10.2 The eluent flow rate may be reduced by 20 - 40 percent to increase separation slightly.

15.10.3 A new initial calibration must be run under these modified analytical conditions prior to sample analysis.

14.3 Initial Calibration

14.3.1 Calibration is performed using the six calibration standards prepared in Sections 12.3 and 12.4, and a blank. The calibration must bracket sample analyte concentrations. The low point of the curve must be at or below the minimum reporting limit.

Note: Calibration and quantitation for bromide must be done by peak height.

- 14.3.2 Set up the IC as shown in Attachment 23.2. Inspect all connections carefully.
- 14.3.3 Start the instrument following the startup instructions given in Section 15. First, run an injection of laboratory reagent water to completion. Then, run the calibration (Sections 12.3.1. and 12.4.1) in succession from low to high concentration.
- 14.3.4 Using peak areas (or height when required) prepare a linear calibration curve. Linear regression must be used for all quantitation. The correlation coefficient (r) must be greater than 0.99 ($r^2 > 0.995$). Refer to Attachment 23.3.
- 14.3.5 After the initial calibration has been run and is acceptable, analyze a second-source calibration verification (SCV) at the mid-point of the calibration. If the recovery is not within $\pm 10\%$, repeat the calibration and SCV.
- 14.4 Calibration Verification
- 14.4.1 Once the initial calibration curve and SCV has been run and is acceptable, analyze the mid-point standard from the calibration curve. This is the initial calibration verification (CCV). If response for any analyte varies from the expected value or retention time by more than $\pm 10\%$, locate the problem and repeat the calibration and calibration verification successfully. Input retention times into retention time window spreadsheet.
- 14.4.2 Analysis of an initial calibration blank (CCB/BLK) must immediately follow the CCV. The BLK is an aliquot of laboratory reagent water. The result for the initial BLK must not exceed three times the method detection limit (MDL). If these criteria cannot be achieved, appropriate corrective action must be taken before samples are analyzed. The initial BLK must be input to the laboratory information management system as a blank. Element™ will not allow entry of a CCV result without an associated CCB/BLK.
- 14.4.3 After the initial CCV/CCB, a contract required detection limit standard (CRDL) is analyzed. The CRDL is an aliquot of the lowest calibration standard analyzed like a sample. Recovery of the CRDL must be within 50 -150% of the expected value.
- 14.4.4 The calibration must be verified during sample analysis using continuing calibration standards, CCV/CCB. CCV/CCB's are analyzed after every ten samples and at the end of the analytical batch. Included in the 10-sample count are all samples and quality control injected after the initial CCB/BLK. If the response for any analyte varies from expected value or retention time by more than $\pm 10\%$, locate the problem and repeat the calibration verification successfully. If results still exceed $\pm 10\%$, sample analysis must be terminated and recalibration is needed. All samples associated with an unacceptable CCV/CCB must be reanalyzed.
- Note: For Department of Defense (DoD) samples, any analyte concentration found in the method blank must be less than one half the reporting limit instead of less than three times the MDL. If this is not achieved for the final report, narrate samples in accordance with Attachment 23.6.
- 14.5 Section 14.4 is repeated on a daily basis to verify the initial calibration. The initial calibration is considered acceptable until the failure of one or more calibration verification requirements.
- 14.6 A new calibration must be prepared and run if stock standards are changed.

15.0 ANALYTICAL PROCEDURE

- 15.1 Follow the sample collection, preservation and pretreatment instructions given in Sections 8.0 and 13.0.
- 15.2 Turn on the power to Dionex RFIC instrument and auto sampler.
- 15.3 Add laboratory reagent water to the eluent reservoir and prime the eluent delivery pump. Be sure that all air bubbles are eliminated from the inlet filter and the eluent inlet tubing. Connect to remote control panel on computer and start eluent flow by clicking test button twice. Refer to Attachment 23.2
- 15.3.1 The test button '27mM/17mA' is for the anions Fluoride, Chloride, Nitrite, Sulfate, and Nitrate. The test button 'Bromide' is for the anions Bromide.
- 15.4 After starting the eluent flow, let the IC stabilize for at least one hour before analysis and conductivity drop below 0.5 μ S.
- 15.5 Prepare 5 mL sample vials by labeling and placing them into vial trays. Pour standards and samples into corresponding vials. Place filter cap on vial (filter down). Using black cap plunger, plunge filter caps down into vials flush with the top of the vial.
- 15.6 Load vial tray into the left side of auto sampler. Vial spot number 1 (indicated on vial tray) should be orientated on the right. Push the RUN/HOLD button on auto sampler to move vials into position for sampling.
- 15.7 Copy and create a run batch from a previous run batch, save as current date (MMDDYY). If using a previous calibration, check raw data box in save window.
- 15.7.1 Set up run order of standards and samples. An initial injection of laboratory reagent water must be run as the first sample of the day. Include CCV/CCB set every ten samples and at the end of the run.
- 15.7.2 The status on samples that need to run should say "Single". The status of a previous calibration will say "Finished".
- 15.8 Once the run batch is set, click the 'start/stop batch' button. In the pop-up window, select batch name to be ran then click start. The first sample in the batch with single status should be highlighted green and the auto sampler should start.
- 15.9 Nonlinear response can result when the analytical column capacity is exceeded (overloading). If any analyte response exceeds the calibration range, dilute the sample with laboratory reagent water and reanalyze. Nonlinear response for fluoride or chloride can also occur if elution is during or very near the signal suppression, caused when water elutes into the detector.

16.0 CALCULATIONS AND DATA HANDLING

- 16.1 Analyte quantitation is performed by regression from the initial calibration curve. Peak area is used by default. Peak height must be used when necessitated by chromatographic interferences and/or analytes. The curve and all standards must be reprocessed using peak height when height is used. Multiply all extracted soil sample results by 10 to compensate for the 1:10 dilution and report as "soluble", not "total".
- 16.2 Report only those values that fall within the calibration range. Samples exceeding the highest calibration standard must be diluted and reanalyzed as directed in Section 15.8.
- 16.3 Report water results in mg/L, report extracted soil results in mg/kg soluble dry.
- 16.4 Report the following anions accordingly:
- 16.4.1 Nitrite (NO_2^-) as N
 - 16.4.2 Nitrate (NO_3^-) as N
 - 16.4.3 o-Phosphate (HPO_4^{2-}) as P

Note: Verify each calculation to convert anion results by peer review.

17.0 DATA REPORTING AND DELIVERABLES

- 17.1 Analysts processing samples are responsible for data quality and for correctly filling in the proper documentation. This is mandatory for quality control purposes, and to provide clients with defensible data.
- 17.2 The following must be attached to the laboratory benchsheet with each batch:
- 17.2.1 Chromatograms containing named peaks of ions being analyzed
 - 17.2.2 Calibration curve printouts for each ion
 - 17.2.3 Copy of calibration standards
 - 17.2.4 Actual run time report for samples analyzed

Note: Refer to the calibration example (Attachment 23.3) and the run logbook example (Attachment 23.4).

- 17.3 The instrument run logbook must be filled in for each batch analyzed with the following information:
- 17.3.1 Date analyzed
 - 17.3.2 Analyst's initials
 - 17.3.3 Method name and number
 - 17.3.4 Calibration standards used with units

17.3.5 Client name and sample numbers analyzed

Note: Refer to the attached instrument logbook example (Attachment 23.4).

17.4 All logbooks must be filled in completely and correctly. All corrections are to be made in indelible ink. **Corrections are to be made with a single lineout, which is then dated and initialed. Write-overs are not acceptable.** Erroneous results must remain legible. Any new result is placed near the incorrect result. Blank lines in the logbook must be Z'd out.

17.5 All completed batch analyses must include the following:

17.5.1 BLK, BS and sample concentrations with units

17.5.2 IMS, MSD, and DUP, results with units

17.5.3 Instrument number, date run, stock standard number

17.5.4 Standard values, observed standard values, working standard numbers

17.5.5 Refer to the attached preparation batch report example (Attachment 23.5).

17.6 If internal chain-of-custody (CoC) is required, it is very important that CoC forms be filled in completely and correctly.

18.0 QUALITY ASSURANCE

18.1 Method and matrix QC must be analyzed for each batch. A batch consists of a maximum of 20 samples per matrix and may be left open for up to 24 hours.

18.2 Method QC consists of a blank (BLK), blank spike (BS), duplicate sample (DUP), continuing calibration verification (CCV), continuing calibration blank (CCB), and a second-source calibration verification (SCV).

18.2.1 A BLK is an aliquot of laboratory reagent water analyzed once per batch of samples.

18.2.2 A BS is an aliquot of laboratory reagent water spiked with a known amount of anion stock standards. Into 10 mL of reagent water, spike 20 µL for Fluoride, Nitrite, Nitrate, and Bromide anions; and 200 µL for Chloride and Sulfate anions. Acceptance limits are listed in Element™. A BS is analyzed once per a batch.

18.2.3 A DUP is a second sample aliquot analyzed the same as the first. This is to be performed every ten samples. The RSD needs to be less than or equal to 20.

18.2.3 A CCV is an aliquot of a mid-point standard. The acceptance limits are 90 - 110% of actual value. A CCV is analyzed at the beginning and end of each run and every 10 readings.

18.2.4 A CCB is an aliquot of laboratory reagent water. A CCB is analyzed after each CCV.

- 18.2.5 A SCV is an aliquot of known concentration, usually mid-calibration range, from the second source calibration standards. Acceptance limits are 90 - 110% of the calculated value. The SCV is analyzed once per calibration.
- 18.2 Matrix QC consists of a matrix spike (MS) and matrix spike duplicate (MSD).
- 18.2.1 A MS is an aliquot of sample spiked with a known amount of anion stock standards. Into 10 mL of filtered sample, spike 20 μ L for Fluoride, Nitrite, Nitrate, and Bromide anions; and 200 μ L for Chloride and Sulfate anions. Acceptance limits are listed in Element™.
- 18.2.1.1 Method 300 At least one MS is required every 10 samples.
- 18.2.1.1 Method 9056A At least one MS is required per batch.
- 18.2.2 A MSD is a duplicate of the MS, spiked and analyzed the same as the MS. The sample volume used for the MSD needs to be the same as used for the MS.
- 18.2.2.1 Method 300 No MSD required
- 18.2.2.2 Method 9056A At least one MSD required per batch.
- 18.3 If any quality control parameter is out of established control limits, initiate corrective action that includes the following:
- 18.3.1 Perform corrective action in accordance with TriMatrix SOP GR-10-106.
- 18.3.2 Once corrective action has been taken, re-analyze all samples associated with the out-of-control event unless otherwise specified
- 18.4 All manual integrations are to be performed and documented in accordance with TriMatrix SOP GR-10-115.
- 19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION**
- 19.1 Before the analysis of 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.
- 19.1.1 Initial Demonstration of Capability
- 19.1.1.1 Spike four aliquots of laboratory reagent water with the SCV standard so concentrations are in the lower half of the calibration range. Process the four spikes following every step of the procedure. Input results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation. Average percent recovery must be between 90 - 110%. Standard deviation of the average 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 measurement system. If this occurs, locate and correct the source of the problem and repeat the study. Samples may not be analyzed by any analyst or on any instrument until an analyst demonstration of capability study has been successfully completed. Copies of all IDC studies attempted, with raw data, must be given to the Quality Assurance department.

19.1.2 A Continuing Demonstration of Capability (CDC) must be performed annually by any of the following approaches:

19.1.2.1 By repeating the IDC study.

19.1.2.2 By exclusively running four consecutive SCVs during the course of routine sample analysis.

19.1.2.3 By exclusively and successfully completing a blind performance testing analysis during the course of routine sample analysis.

19.1.2.4 By using the last four of seven MDL study results if run exclusively by the analyst. ONLY the last four results may be used.

Note: Copies of the all CDC studies and raw data must be given to the Quality Assurance department.

19.2 A Method Detection Limit Study (MDL) is required every six months in accordance with TriMatrix 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 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 *EMSL, Office of Research and Development, "Determination of Inorganic Anions by Ion Chromatography". USEPA Method 300.0, USEPA, Cincinnati, OH 45268, USA, rev. 2.1, 1993*
- 22.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Revision 1, February 2007, Method 9056A, "Determination of Inorganic Anions by Ion Chromatography"*

23.0 ATTACHMENTS

- 23.1 Retention Time Spreadsheet
- 23.2 Control Panel for the Dionex 2000
- 23.3 Calibration Example
- 23.4 Instrument Number 306 Run Logbook Example
- 23.5 Preparation Batch Report Example
- 23.6 Element™ Data Qualifiers (SQD)

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Attachment 23.1
 Retention Time Spreadsheet



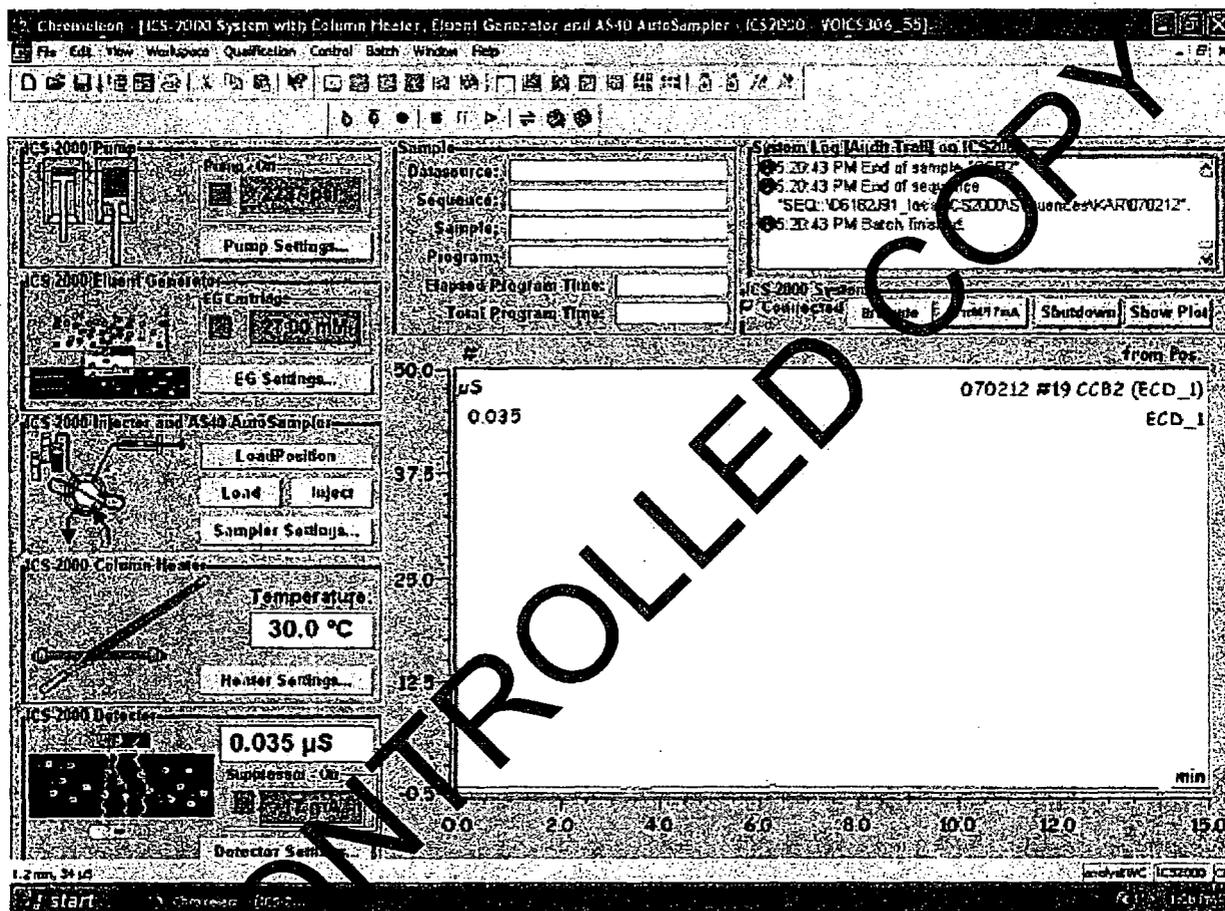
			Chloride				Fluoride				Nitrate			
Date	time	std ID	conc	ret tim	LCL _{RT}	UCL _{RT}	conc	ret tim	LCL _{RT}	UCL _{RT}	conc	ret tim	LCL _{RT}	UCL _{RT}
06/18/12	16:03	cal 1	0.00				0.00				0.00			
06/18/12	16:21	cal 2	1.00	5.03	4.95	5.09	0.05	3.77	3.66	3.87	0.05	9.344	9.28	9.41
06/18/12	16:38	cal 3	2.00	5.01	4.93	5.07	0.10	3.747	3.64	3.85	0.10	9.317	9.25	9.39
06/18/12	16:56	cal 4	8.00	4.994	4.92	5.06	0.40	3.747	3.64	3.85	0.40	9.264	9.20	9.33
06/18/12	17:13	cal 5	20.00	4.994	4.92	5.06	1.00	3.75	3.65	3.86	0.10	9.204	9.14	9.27
06/18/12	17:30	cal 6	60.00	4.987	4.91	5.05	3.00	3.67	3.56	3.87	3.00	9.064	9.00	9.13
06/18/12	17:48	cal 7	100.00	4.973	4.90	5.04	5.00	3.7	3.6	3.85	5.00	8.963	8.89	9.03
0.12-05.1	20:56	RT 1	60.00	4.964			3.00	3.797			3.00	9.16		
0.12-05.1	4:46	RT 2	60.00	4.984			3.00	3.877			3.00	9.177		
0.12-05.1	10:36	RT 3	60.00	4.977			3.00	3.827			3.00	9.17		
06/22/12	10:13	CCV	60.00	4.99			3.00	3.77			3.00	9.06		
			Nitrite				Sulfate				Bromide			
Date	time	std ID	conc	ret tim	LCL _{RT}	UCL _{RT}	conc	ret tim	LCL _{RT}	UCL _{RT}	conc	ret tim	LCL _{RT}	UCL _{RT}
06/18/12	16:03	cal 1	0.00				0.00							
06/18/12	16:21	cal 2	0.05	5.91	5.84	5.98	1.00	7.477	7.43	7.58	0.05			
06/18/12	16:38	cal 3	0.10	5.89	5.81	5.95	2.00	7.484	7.44	7.58	0.10			
06/18/12	16:56	cal 4	0.40	5.874	5.80	5.94	8.00	7.427	7.38	7.53	0.40			
06/18/12	17:13	cal 5	1.00	5.86	5.79	5.93	20.00	7.334	7.29	7.43	1.00			
06/18/12	17:30	cal 6	3.00	5.844	5.77	5.91	60.00	7.144	7.10	7.24	3.00			
06/18/12	17:48	cal 7	5.00	5.817	5.74	5.88	100.00	7.017	6.97	7.12	5.00			
0.12-05.1	20:56	RT 1	3.00	5.857			60.00	7.27			3.00			
0.12-05.1	4:46	RT 2	3.00	5.874			60.00	7.25			3.00			
0.12-05.1	10:36	RT 3	3.00	5.867			60.00	7.254			3.00			
06/22/12	10:13	CCV	3.00	5.85			60.00	7.12			3.00			

SOP Name: Determination of Inorganic Anions by Ion Chromatography
EPA Method 300.0, SW-846 Method 9056A
SOP Number: GR-02-113

Revision Number: 3.0
Date Revised: 8/3/12
Date Initiated: 4/1/00

page 17 of 21

Attachment 23.2
Control Panel for the Dionex 2000



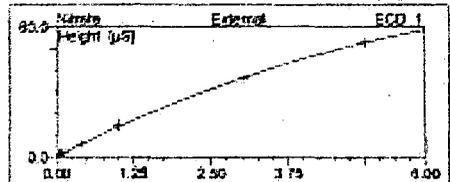
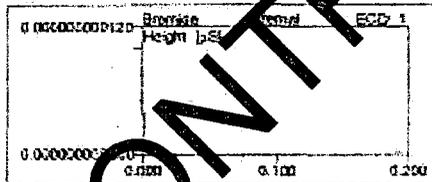
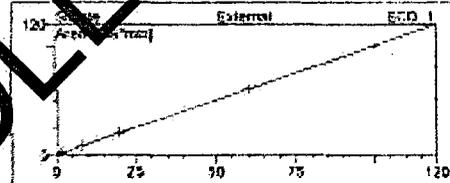
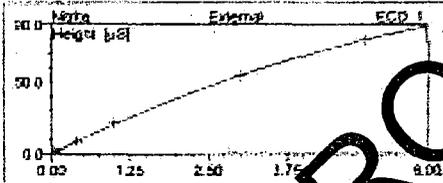
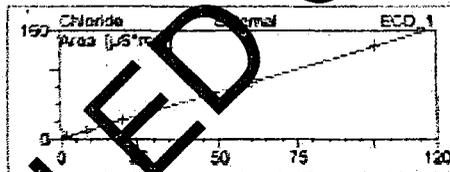
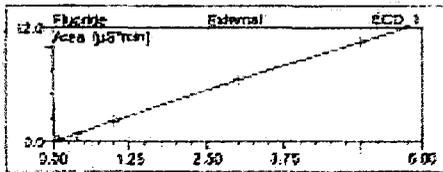
Attachment 23.3
 Calibration Example

Operator: analystWC Timebase: ICS2000 Sequence: 070212

Page 1-1
 7/3/2012 1:10 PM

7 STD B

Sample Name:	STD B	Injection Volume:	25.0
Vial Number:	7	Channel:	ECD_1
Sample Type:	standard		
Control Program:	AS18_2mm		
Quantil Method:	EPA 300.0/SW846-9056	Division Factor:	1.0000
Recording Time:	18.08.12 17:30	Sample Weight:	1.0000
Run Time (min):	15.00	Sample Amount:	1.0000



No.	Ret. Time (min)	Peak Name	Cal. Type	Points	Coeff. Det. %	Offset	Slope	Curve
2	2.77	Fluoride	QOff	6	99.9918	-0.0270	2.2543	-0.0312
3	4.99	Chloride	LOff	7	99.9582	0.1184	1.3544	0.0000
4	5.34	Nitrite	QOff	6	99.9869	0.1959	21.3487	-1.1030
5	7.14	Sulfate	LOff	7	99.9944	0.2237	1.0010	0.0000
7	9.06	Nitrate	QOff	6	99.9814	0.1606	14.3933	-0.7781
Average:					99.9905	0.1343	8.0723	-0.3825

SOP Name: Determination of Inorganic Anions by Ion Chromatography
EPA Method 300.0, SW-846 Method 9056A
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Revision Number: 3.0
Date Revised: 8/3/12
Date Initiated: 4/1/00

Attachment 23.4
Instrument Number 306 Run Logbook

TRIMATRIX
LABORATORIES
Instrument Number 306
Dionex ICS-2000

Date: 7/2/12 Quantitation Method: AS18 Analytical Column #: 1050923 Curve Date: 6/18/12
System Operator Initials: KRE Program Method: AS18.dmm Guard Column #: 2040930 Sample Name: 070212

Sample Name	Element Number	Client	Method Reference Number	Dilution Prep		CCV Pass / Fail (90)	Notes, Standard #'s, etc.
				Sample Volume	Final Volume		
R		QC	3000/1000				
CCV1							P 2070125
CCB1/BIK							
BS							2070127
BSD							2070127
CRD							2070126
LDD	1201380-17	Trimatrix					
LDD	1204008-17						
CCV2		QC					P 2070125
CCB2							
							KRE 7/3/12

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SOP Name: Determination of Inorganic Anions by Ion Chromatography
 EPA Method 300.0, SW-846 Method 9056A
 SOP Number: GR-02-113 page 20 of 21

Revision Number: 3.0
 Date Revised: 8/3/12
 Date Initiated: 4/1/00

Attachment 23.5
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH **0609404** Page 1 of 1

Printed: 10/29/2007 5:01:45PM

Inorganic - Wet Chemistry, Water, General Inorganic Prep
 (No Surrogate)

Batch Comments: (none)

<u>Work Order</u>	<u>Analysis</u>	<u>Work Order</u>	<u>Analysis</u>	<u>Work Order</u>	<u>Analysis</u>
0608181	Chloride 300.0				

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0609404-BLK1		Aug-14-06 15:00	LEW	10	10					BLENK	
0609404-BS1		Aug-14-06 15:00	LEW	10	10			A608419	100	LCS	
0609404-MS1		Aug-14-06 15:00	LEW	10	10		0608181-01	A608412	100	MATRIX SPIKE	
0609404-MSD1		Aug-14-06 15:00	LEW	10	10		0608181-01	A608412	100	MATRIX SPIKE DUP	
0608181-01	A	Aug-14-06 15:00	LEW	10	10						
0608181-02	A	Aug-14-06 15:00	LEW	10	10						
0608181-04	A	Aug-14-06 15:00	LEW	10	10						
0608181-05	A	Aug-14-06 15:00	LEW	10	10						
0608181-06	A	Aug-14-06 15:00	LEW	10	10						
0608181-07	A	Aug-14-06 15:00	LEW	10	10						
0608181-08	A	Aug-14-06 15:00	LEW	10	10						
0608181-09	A	Aug-14-06 15:00	LEW	10	10						
0608181-10	A	Aug-14-06 15:00	LEW	10	10						
0608181-11	A	Aug-14-06 15:00	LEW	10	10						
0608181-12	A	Aug-14-06 15:00	LEW	10	10						

Comments:	Analyst Initials:

**Attachment 23.6
 Element™ Data Qualifiers for Method Blanks (DoD)**

Element Data Qualifiers (DoD Method Blanks)		Equations		
Qualifier #	Qualifier	Method Blank (MB) Conditional	Sample (S) Conditional	Action Required
DoD10	The analyte concentration in the associated MB was greater than or equal to 1/2 the RL. The positive sample result, which was less than 5 times the MB value, is considered estimated.	$MB \geq 0.5 \cdot RL$	$S < 5 \cdot MB$	estimate
DoD11	The analyte concentration in the associated MB for this common lab contaminant was greater than or equal to the RL. The positive sample result, which was less than 5 times the MB value, is considered estimated.	$MB \geq 0.5 \cdot RL$	$S < 5 \cdot MB$	estimate
DoD12	The analyte concentration in the MB for this common lab contaminant was greater than or equal to the RL. All associated samples had results less than the RL. No qualification is required.	$MB \geq RL$	$S < RL$	none
DoD13	The analyte concentration in the MB for this common lab contaminant was greater than 1/2 the RL, but less than or equal to the RL. All associated samples had results less than the RL. No corrective action is required.	$0.5 \cdot RL < MB < RL$	$S < RL$	none
DoD14	The analyte concentration in the associated MB was greater than or equal to 1/2 the RL. The positive sample result, which was greater than 5 times the MB value, is not considered estimated.	$MB \geq 0.5 \cdot RL$	$S > 5 \cdot MB$	none
DoD15	The analyte concentration in the MB for this common lab contaminant was greater than 1/2 the RL, but less than the RL. The positive sample result, which was less than 5 times the MB value, is considered estimated.	$0.5 \cdot RL < MB < RL$	$S < 5 \cdot MB$	estimate
DoD22	The analyte concentration in the associated MB was greater than or equal to 1/2 the LOQ. The positive sample result, which was less than 10 times the MB value, is considered estimated.	$MB \geq 0.5 \cdot LOQ$	$S < 10 \cdot MB$	estimate
DoD23	The analyte concentration in the associated MB for this common lab contaminant was greater than or equal to the LOQ. The positive sample result, which was less than 10 times the MB value, is considered estimated.	$MB \geq LOQ$	$S < 10 \cdot MB$	estimate
DoD24	The analyte concentration in the MB for this common lab contaminant was greater than or equal to the LOQ. All associated samples had results less than the LOQ. No qualification is required.	$MB \geq LOQ$	$S < LOQ$	none
DoD25	The analyte concentration in the MB for this common lab contaminant was greater than 1/2 the LOQ, but less than or equal to the LOQ. All associated samples had results less than the LOQ. No corrective action is required.	$0.5 \cdot LOQ < MB < LOQ$	$S < LOQ$	none
DoD26	The analyte concentration in the associated MB was greater than or equal to 1/2 the LOQ. The positive sample result, which was greater than 10 times the MB value, is not considered estimated.	$MB \geq 0.5 \cdot LOQ$	$S > 10 \cdot MB$	none
DoD27	The analyte concentration in the MB for this common lab contaminant was greater than 1/2 the LOQ, but less than the LOQ. The positive sample result, which was less than 10 times the MB value, is considered estimated.	$0.5 \cdot LOQ < MB < LOQ$	$S < 10 \cdot MB$	estimate
DoD28	The analyte concentration in the associated MB for this common lab contaminant was greater than or equal to the LOQ. The positive sample result, which was greater than 10 times the MB value, is not considered estimated.	$MB \geq LOQ$	$S > 10 \cdot MB$	none

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STANDARD OPERATING PROCEDURE

Volatile Organic Compounds

EPA Methods 601 and 602
SW-846 Methods 8021B and 5030B

APPROVALS:

Area Supervisor: *Diane VanMale* Date: 2-20-12
Diane L. VanMale

QA Officer: *Tom C. Booche* Date: 2-17-12
Tom C. Booche

President: *Douglas E. Kriscunas* Date: 2-20-2012
Douglas E. Kriscunas

Procedure Number: GR-03-105
Revision Number: 6.3

Date Initiated: 1/27/05
Effective Date: 2/28/12

Date Revised: 2/17/12
Pages Revised: All

By: Tom C. Booche

Total Number of Pages: 39

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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 is a purge and trap capillary column gas chromatography (GC) method used to determine volatile organic compounds in a variety of matrices. This procedure is applicable to nearly all sample matrices including groundwater, municipal and industrial discharges, 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. A second analytical technique (GC/MS) is used when needed to confirm reportable measurements.
- 1.2 Analytes routinely analyzed by TriMatrix Laboratories are listed with the corresponding reporting limit in Attachment 23.1, Table 1. Actual reporting limits will be higher for highly concentrated samples requiring dilution and/or methanol extraction preparation, or for samples with low percent solids. The applicable concentration range is instrument and compound dependent but is generally 1.0 to 200 ug/L for water samples and 0.01 to 100 mg/kg for soil/solid samples.
- 1.3 Two of the isomeric xylenes are not resolved on the capillary column and must be reported as an isomeric pair (meta-xylene and para-xylene).
- 1.4 This procedure is used when analyzing volatile compounds in accordance with methods 601, 602 and 8021B. The GC system may be set up so a single detector is used when only analyzing for aromatics (602/8021B) or halogenates (601/8021B).
- 1.5 This procedure outlines in detail where the methods 601/602 differ from method 8021B. Wastewater analysis for effluent reporting must be in accordance with method 601/602.

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 8021B, "Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors", Revision 2, December, 1996*
- 2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, December 1996, Method 5030B, "Purge-and-Trap for Aqueous Samples", Revision 2, December, 1996*
- 2.3 *Code of Federal Regulations, Title 40 Protection of Environment, Volume 19, July 1, 2001 Edition, Chapter I Environmental Protection Agency, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Purgeable Halocarbons, Method 601*
- 2.4 *Code of Federal Regulations, Title 40 Protection of Environment, Volume 19, July 1, 2001 Edition, Chapter I Environmental Protection Agency, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Purgeable Aromatics, Method 602*

3.0 SUMMARY OF PROCEDURE

- 3.1 An inert gas (helium) is bubbled through a 5 mL water sample, a 5 g soil sample or a 5 mL water sample containing an aliquot of a soil/methanol extract, transferring the purgeable organics from the aqueous phase to the vapor phase. The vapor phase is swept through a sorbent trap where

the purgeables are absorbed. After purging, the trap is backflushed with the inert gas to desorb the compounds onto a gas chromatographic column.

3.2 After introduction, the gas chromatograph is temperature programmed to separate the organic compounds of interest using a capillary column. Reporting is achieved by either a photoionization detector (PID) and/or an electrolytic conductivity detector (ELCD) in series. The PID is used for detecting aromatic compounds and compounds with double bonds, while the ELCD is used for detecting halogenated compounds. Tentative identifications are obtained by analyzing standards under the same conditions used for samples and comparing analyte retention times. Concentrations are measured by relating the response produced for that compound to the response produced by a compound that is used as an internal standard.

3.3 This method can be used for most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Water-soluble volatiles may be analyzed by this technique, however, quantitation limits will be higher than those listed in Attachment 23.1 - Table 1.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to Table 1

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-3-124, *Volatile Laboratory Corrective Actions*, latest revision

5.3 TriMatrix SOP GR-1-102, *Laboratory Waste Disposal*, latest revision

5.4 TriMatrix SOP GR-10-123, *Element™ Data Transfer and Review*, latest revision

5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Whether purged or injected directly on the column, interferences naturally present in the samples can cause elevated reporting limits and interfere with the analysis. Naturally occurring interferences can vary considerably from site to site.

6.2 Sample contamination can also cause raised reporting limits or false positives, and can come from a variety of sources. Improper sampling techniques can contaminate the sample at the job site. During shipment and storage, volatile organics (particularly methylene chloride and fluorocarbons) can diffuse through the septum and into the sample. A trip blank prepared from reagent water and carried through the sampling and handling protocol will serve as a check on such contamination.

- 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 where they were deposited by a previously analyzed high-level sample. To reduce this, the use of non-PTFE plastic tubing, non-PTFE thread sealants, and flow controllers with rubber components in the purging device have been eliminated where possible.
- 6.4 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe is rinsed with reagent water between samples. Where practical, samples with unusually high concentrations should be followed by an analysis of reagent water to check for cross contamination. If the compounds present in high concentrations are also present in subsequent samples, the analyst must demonstrate that the positive results are not due to carryover by reanalysis of the samples. However, if the compounds are not present in the subsequent sample, then reanalysis is not necessary.
- 6.5 The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required. If the contamination is persistent, the complete purge and trap system should be purged first with 100° C reagent water, then, if necessary, with methanol. If methanol is used, either disconnect the trap or install a blank trap, as the methanol will adversely affect the trap's performance.
- 6.6 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105° C oven between analyses.
- 7.0 SAFETY PRECAUTIONS**
- 7.1 Wear a laboratory coat and approved safety glasses while in the 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.
- 7.4 The acute 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 exposure 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 Waste samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-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 Sample Collection

- 8.1.1 Collect all aqueous and liquid waste samples in duplicate 40 mL borosilicate glass screw-cap VOA vials with PTFE-lined silicone septa. Gently fill sample bottles to overflowing to minimize loss of volatiles. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed. Invert the vial to confirm that there is no headspace present. If there is headspace or bubbles larger than 5-6 mm in diameter, refill a new preserved vial.
- 8.1.2 Collect soil/sludge/solid waste samples in accordance with USEPA Method 5035, as specified in TriMatrix SOP GR-04-105, *Closed System Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples*, latest revision.

8.2 Sample Preservation

8.2.1 Aqueous Samples

- 8.2.1.1 For aqueous samples, if the sample contains residual chlorine, collect the sample in a 125 mL container which has been pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl to mix sample and transfer to a 40 mL VOA vial. Cool to 0 - 6° C and adjust pH to <2 with HCl. EPA Methods 330.4 and 330.5 may be used for measurements of residual chlorine. Field test kits are also available for this purpose.
- 8.2.1.2 TriMatrix Laboratories supplies all clientele with 40 mL VOA vials which are pre-preserved with HCl. If the vials were not supplied by the laboratory, and are not pre-preserved, fill vials and adjust the pH to <2 by carefully adding two drops of 1:1 HCl to each sample. Seal the sample bottle, PTFE-face down, and cool to 0 - 6° C.
- 8.2.1.3 For samples requiring analysis by EPA Method 602, if samples are received unpreserved, the hold time is 7 days from the date and time of collection.
- 8.2.1.4 For samples requiring analysis by EPA Method 601, the samples may be collected in unpreserved vials. The hold time will remain at 14 days from the date of collection.

8.2.2 Soil/Sludge/Waste Samples

- 8.2.2.1 The preservation techniques for soils, sludges, and wastes are detailed in TriMatrix SOP number GR-04-105, *Closed System Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples*, most current version.

8.3 Trip Blanks - Aqueous Samples Only

8.3.1 A trip blank should accompany each sample set into the field and back. A sample set is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill a pre-preserved sample bottle with reagent water, seal, and ship to the sampling site along with empty sample bottles. Wherever a set of samples is shipped and stored, it is accompanied by the appropriate trip blank.

8.4 Sample Storage

8.4.1 The samples must be chilled to 0 - 6° C on the day of collection and maintained at that temperature until analysis. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be at 0 - 6° C upon arrival to the laboratory.

8.4.2 Store samples at 0 - 6° C until analysis. The laboratory sample storage area must be free of organic solvent vapors. Soils, sludges, and wastes are stored separately from aqueous samples. To monitor potential cross-contamination, storage blanks will be put in each refrigerator used for sample storage. The storage blanks will be replaced and analyzed by the GC/MS lab on a weekly basis.

8.4.3 Analyze all samples within 14 days of collection. Samples not analyzed within this period must be qualified as estimated. The only exceptions to this rule are for samples for EPA Methods 601 and 602 as detailed in Section 8.2.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Glassware and Hardware

- Class A volumetric flasks- 10 mL, 50 mL, 100 mL, 250 mL, 500 mL
- Various size micro syringes - 2 µL, 10 µL, 100 µL, 1000 µL
- 5 mL Luerlock syringes
- 60 or 125 mL wide mouth septum or PTFE-lined jar
- 40 mL PTFE-lined, screw-cap septum vials
- 20 mL PTFE-lined, screw-cap septum vials
- 4 mL PTFE-lined, screw-cap septum vials (for transfer of methanol extracts)
- refrigerator
- pH test strips
- thin metal spatulas

- analytical balance - 0.0001 g
- top-loading balance - 0.01 g
- pasteur pipettes (disposable)
- pipette bulb

9.2 Instrumentation

9.2.1 Autosamplers

- O. I. Analytical 4552 (Archon)
- Dynatech PTA 30 W/S

9.2.2 Concentrators

- Tekmar LSC-2000 and EST Encon

Recommended conditions:

- Trap: Vocarb 3000 (other traps may be substituted if demonstrating acceptable performance)
- Purge Gas: Helium @ 35-40 mL/minute
- Purge 11 minutes @ ambient temperature (45° C heated purge for soil samples)
- Desorb 2 minutes @ 250° C
- Bake 10 minutes at 260° C

9.2.3 Gas Chromatograph

- Hewlett Packard 5890 II and Agilent 6890 gas chromatographs equipped with Photoionization Detector (PID) and Hall Electrolytic Conductivity Detector (ELCD) in series.

Current Conditions:

- Injector Temperature: 200° C
- PID Detector Temperature: 220° C
- ELCD Reactor Temperature: 900-980° C
- ELCD Reactor Base Temperature: 220° C
- ELCD Reaction Gas: Hydrogen 60 mL/minute

- ELCD Solvent: n-Propanol 35 μ L/minute

9.2.4 Current Columns and Conditions (See Attachment 23.3, Table 3 – Elution Order on DB-VRX):

- 75m x 0.45 mm ID wide-bore capillary column, 1.55 μ m film thickness (J&W Scientific DB-VRX or equivalent)
- Temperature Program: 35° C for 12 minutes, then ramp to 60° C @ 5°/minute, hold 1.0 minute, ramp 17°/minute to 100° hold 3 minutes, ramp 1°/minute to 225° hold 4 minutes.
- Carrier Flow: Helium @ 10 mL/minute
- Make up flow: Helium @ 35 mL/minute (45 mL/minute total)
- See Attachment 23.7, Figure 4 – Example Chromatogram on DB-VRX (HECD detector) Standard List and Attachment 23.8, Figure 5 – Example Chromatogram on DB-VRX (PID detector) Standard List.

9.2.5 Alternate Column (for aromatic analysis only)

- 30m x 0.53 mm ID wide-bore capillary column, 3 μ m film thickness (J&W Scientific DB-624 or equivalent)
- Temperature Program: 35° then ramp at 6.5°/minute to 110°, 20°/minute to 220°, hold 2 minutes
- Carrier Flow: 10 mL/minute
- Make up Flow: 30 mL/minute (40 mL/minute total)
- See Attachment 23.6, Figure 3 – Example Chromatogram on DB-624 (PID detector) Limited Aromatics List.

9.2.6 Data Acquisition

- PE Nelson Turbochrom Data Acquisition System

Note: Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

Note: Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Gas Chromatographs

- 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

10.2 Detectors

- 10.2.1 Check detector signals daily
- 10.2.2 Clean or replace PID lamps as needed
- 10.2.3 Check and refill ELCD solvent reservoir daily
- 10.2.4 Replace ELCD resin cartridge every 3 months
- 10.2.5 Replace ELCD reaction tube as needed
- 10.2.6 Backflush and replace ELCD transfer line if particulates accumulate

10.3 Purge and Trap

- 10.3.1 Bake out trap daily before use
- 10.3.2 Check purge flow weekly
- 10.3.3 Empty waste bottles and fill rinse bottles daily
- 10.3.4 Check concentrator pressures
- 10.3.5 Check and fill internal/surrogate standard syringe daily

11.0 CHEMICALS AND REAGENTS

11.1 Chemicals

- Reagent water (organic free) – produced from double-distilled reverse osmosis fed water, followed by purging with helium for 30 minutes.
- Methanol (purge and trap grade)
- Pure stock standard materials or certified stock standards (96% pure or greater)
- 1:1 Hydrochloric Acid

- Sodium bisulfate, NaHSO₄ - ACS reagent grade or equivalent

12.0 STANDARDS PREPARATION

12.1 All standards used in the laboratory must be recorded in Element™. Standard ID, vendor, part number, lot number, concentration, solvent, purity are recorded. Each standard is given a unique ID number. If the standard used is a commercially prepared standard, the certificate of analysis must be kept on file to ensure standard traceability.

12.2 Stock & Intermediate Standard Storage Requirements

12.2.1 Gas standards should be monitored closely for degradation. Stock standards for gases usually will need to be replaced after one month. Intermediate gas standards usually will need to be replaced after one week. If the continuing calibration standard varies by more than 15% from the initial calibration curve, then a new gas standard needs to be prepared. The first compound to fail will generally be dichlorodifluoromethane, so it's response should be monitored closely.

12.2.2 All other stock standards may be kept up to 6 months or as recommended by manufacturer. Intermediate standards may also be kept for up to 6 months; however, they should be continuously monitored for degradation by comparison of the response of the compounds in daily continuing calibration standards to that of the initial calibration curve.

12.2.3 2-Chloroethyl-vinyl ether should be prepared separately from the other compounds due to its reactivity. Intermediate standards should be prepared fresh monthly.

12.2.4 All stock and intermediate standards are to be stored with minimal headspace at -10° C to -20° C.

12.3 Stock Standards

12.3.1 Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source. The stock standards used for calibration and the laboratory control standard are purchased at a concentration of 2000 ug/mL.

12.3.2 If preparing stock standards from neat compounds, no correction factor is needed when the chemical has a purity of greater than or equal to 96%. If the purity is less than 96%, the concentration must be corrected to calculate the true value. Prepare stock standard solutions in methanol using assayed liquids. Transfer the stock standard solution into PTFE-sealed screw-cap bottles. Store in a freezer separate from samples and protect from light. The 2-chloroethyl-vinyl ether and internal and surrogate spiking standards are prepared from neat compounds.

12.3.2.1 Gravimetric Method

10,000 ug/mL stock standard: Weigh 0.5 g of each analyte (neat) into a 50 mL volumetric flask partially filled with methanol which has been tared on an analytical balance. The weight is recorded to the nearest 0.0001 g. The volumetric is then diluted to volume with methanol, capped, and inverted three times to ensure proper mixing. To estimate the approximate volume of neat compound to inject use the following equation:

$$\left(\frac{1}{\text{density}} \right) \times 500 = \text{uL of analyte of interest}$$

12.3.2.2 Stock Standards for Internal/Surrogate Standard Mixture

Prepare a mixture of Fluorobenzene and 2-Bromo-1-chloropropane (internal standards), along with α, α, α -Trifluorotoluene, 1,2-Dichloroethane-d₄, (surrogate standards), at 10,000 ug/mL each as in 12.3.2.1 above.

Note: If only aromatics are to be analyzed, Fluorobenzene may be used as the only internal standard, since 2-Bromo-1-chloropropane is not detected on the PID.

12.4 Intermediate Standards

12.4.1 200 ug/mL Intermediate Stock Standard

This standard is prepared in methanol by diluting an aliquot of a purchased certified solution into a 1 mL mini-inert vial. Fill the vial with 900 μ L of purge and trap grade methanol, then add 100 μ L of the 2000 ug/mL certified stock standard. If a neat stock standard is used, inject 20 μ L of each 10,000 ug/mL stock standard (12.3.2.1) into the mini-inert vial. Invert 3 times to mix.

12.4.2 250 ug/mL Internal/Surrogate Standard

This standard is prepared by diluting an aliquot of the 10,000 ug/mL neat stock standard into a 100 mL volumetric flask.

12.4.2.1 Prepare a working standard at 250 ug/mL by injecting 2.50 mL of the 10,000 ug/mL stock internal/surrogate standard (Section 12.3.2.2) into a 100 mL volumetric flask partially filled with methanol. Dilute to the mark with methanol, stopper and invert 3 times to mix. The Archon and PTA 30 W/S autosamplers will automatically add 1 μ L to each 5 mL sample aliquot prior to purging.

12.5 Working Standards

12.5.1 Working standards are prepared in water in either a 50 mL volumetric or a 5 mL syringe using the formula below:

$$\left(\frac{C_f \times V_f}{C_s} \right) = V_s$$

where:

V_s = volume of stock to inject (μL)
 C_f = final concentration of working standard ($\mu\text{g/L}$)
 V_f = final volume of working standard (μL)
 C_s = concentration of stock ($\mu\text{g/L}$)

Working standards are prepared as needed and are not retained.

12.6 Laboratory Control Standard (LCS) - 100 $\mu\text{g/mL}$

12.6.1 This standard is prepared in methanol by diluting an aliquot of a purchased certified solution into a 1 mL mini-inert vial. Fill the vial with 900 μL of purge and trap grade methanol, then add 50 μL of the 2000 $\mu\text{g/mL}$ certified stock standard.

12.6.2 The LCS standard must be prepared from a source different than that used for the initial calibration and will be used to verify the acceptance of the initial calibration. This solution will also be used for matrix spikes.

13.0 SAMPLE PREPARATION

13.1 Aqueous Samples:

With the Dynatech PTA 30 W/S and D.I. 4532 autosamplers the samples will essentially need no preparation - the 40 mL vial will be loaded directly into the autosampler. The autosampler will take a 5 mL aliquot of the sample, add 1.0 μL of internal/surrogate standards, and transfer the sample to the sparge vessel prior to initiating the purging sequence. If a dilution is required, prepare the dilution by adding an appropriate aliquot of the sample to a 50 mL volumetric flask $\frac{3}{4}$ full of reagent water and bring to volume. Cover the flask with parafilm and invert while swirling. Repeat two more times. Transfer the diluted sample to a new 40 mL vial and place on the autosampler for analysis. Since this process of taking an aliquot destroys the validity of the sample for future analysis, if only one sample vial has been provided, the analyst should immediately place the remaining sample in a 20 mL vial with no headspace, to be used if subsequent analysis is necessary.

13.1.1 Matrix spikes will be prepared by combining sample aliquots to a 100 mL volumetric flask and filling to approximately $\frac{3}{4}$ full. Add 15 μL of the 200 $\mu\text{g/mL}$ intermediate standard, and bring to volume with additional sample. Cover with parafilm, invert and swirl three times. Transfer the spiked sample to two new 40 mL vials and add to the autosampler for analysis.

13.2 Soil/Sludge and Waste Samples:

13.2.1 Low Concentration Method: This is designed for samples containing individual purgeable compounds of <1 mg/kg. TriMatrix employs two methods for the low-level soils. The closed system purge and trap by EPA method 5035 is based on purging a heated 5-gram sample collected in a vial pre-preserved with a sodium bisulfate solution. This procedure is described in detail in TriMatrix SOP GR-04-105. The vial will be loaded directly onto the autosampler for analysis. The other method, based on

the older 5030B method preparation is detailed below. It is based on purging a heated sample mixed with organic-free reagent water containing the internal/surrogate standard and, if applicable, matrix spiking standards. Analyze all blanks, spikes, standards, and samples under the same conditions as the samples.

13.2.1.1 Use a 5 g sample if the expected concentration is <0.2 mg/kg or a 1 g sample for expected concentrations between 0.1 and 1 mg/kg.

13.2.1.2 A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low concentration method. Follow the initial and daily calibration instructions, except for the addition of a 45° purge temperature.

13.2.1.3 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in section 13.2.1.1 directly into a new 40 mL vial containing a PTFE-coated magnetic stirrer. Note and record the actual weight to the nearest 0.1 g. Add 5 mL of reagent water using a 5 mL Luerlock syringe to the sample and cap the vial. Transfer the vial to the autosampler for analysis.

13.2.1.4 The remaining following steps are the same for both the 5030B and 5035 prepared samples. The autosampler will automatically add an additional 5 mL of water, along with 1 µL of the IS/SS standard to the sample container before purging. Heat the sample to 45° C while purging. Refer to section 9.2.2 for recommended purge and trap procedures.

13.2.1.5 For matrix spikes for samples prepared via Method 5030B, add 1.0 µL of the 200 µg/mL intermediate stock standard to a 5 mL of reagent water prior to adding the water to the sample vial. For samples prepared via Method 5035, add the matrix spike to the sample by inject 1.0 µL of the 200 µg/mL intermediate standard through the septa and into the sodium bisulfate layer. This will give a final spike concentration of 0.040 µg/kg. Spiking amounts are subject to change.

13.2.2 High Concentration Method: The method is based on extracting the soil/sludge with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. An aliquot of the extract is added to organic-free reagent water. This is purged at ambient temperature. All samples with an expected concentration of >1.0 mg/kg should be analyzed by this method. For samples prepared by SW-846 Method 5035, refer to TriMatrix SOP number GR-04-105, *Closed System Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples*. For samples prepared by SW-846 Method 5030B, follow the procedure detailed below.

13.2.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and waste that are insoluble in methanol, weigh 10 g (wet weight) of sample into a tared 20 mL vial. Use a top loading balance. Note and record the actual weight to 0.1 g. For waste that is soluble in

methanol, weigh 1 g (wet weight) into a tared 20 mL vial or a 10 mL volumetric flask.

- 13.2.2.2 For sediment/soil or solid waste, quickly add 10 mL of methanol to the vial. For a methanol miscible sample, dilute the sample to 10 mL with the methanol. Cap and shake for 2 minutes.

Note: If the sample is from the State of Michigan, it must also be sonicated for 20 minutes in an ultrasonic bath after shaking.

Note: Sections 13.2.2.1 and 13.2.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

- 13.2.2.3 After settling has occurred, transfer approximately 4 mL of the extract into a vial for storage, using a disposable pipet. The remainder may be discarded. These extracts should be stored at 0 - 6° C in the dark, prior to analysis.

- 13.2.2.4 For each batch of samples prepped the analyst must also prepare a Method Preparation Blank (MPB) and a Laboratory Fortified Blank (LFB). The MPB will consist of 50 mL of reagent water spiked with 1.0 mL of methanol. The LFB is prepared by spiking 50 mL of reagent water with 15 µL of the 200 µg/mL intermediate standard, along with 985 µL of methanol. The LFB concentration will be 2.0 mg/kg.

- 13.2.2.5 If a screening procedure was followed, use the estimated concentration to determine the appropriate volume of methanol extract to add to the 5 mL of organic-free reagent water for analysis. Otherwise, estimate the concentration range of the sample from the low concentration analysis to determine the appropriate volume. If the sample was submitted as a high concentration sample, start with 1 mL. All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

- 13.2.2.6 Add 1.0 mL of the sample to a 50 mL volumetric flask ¾ full of reagent water, and bring to volume. Cover with parafilm, and invert and swirl three times. Transfer to a new 40 mL vial and place on the autosampler for analysis. The sample will be analyzed under the same conditions as the aqueous samples. Refer to Section 9.2.2 for recommended purge and trap procedures.

- 13.2.2.7 Matrix spikes prepared by spiking 50 mL of reagent water with 15 µL of the 200 µg/mL intermediate standard, along with 1.0 mL of the methanol extracted sample. The matrix spike concentration will be 2.0 mg/kg.

14.0 CALIBRATION PROCEDURES

14.1 Initial Calibration – internal standard calibration will be used to calibrate the analytes in this procedure

14.1.1 Daily Blank: For Dynatech PTA 30 and O.I. 4552 autosamplers, place a 40 mL vial into the autosampler. The autosampler will automatically add the desired amount of IS/SS standard. If the blank shows a contamination peak, reagent water must be helium purged and the blank rerun until the system is contaminant free. Sample analysis may not begin until the system has been demonstrated to be free of contamination by the successful analysis of a clean blank.

14.1.2 Standards: Seven standards will be used in the generation of the calibration curve. One of the standards should be at a concentration at or below the reporting limit. The other concentrations should define the working range of the detector. All target compounds must be included in the calibration curve. The autosampler will add a constant amount of internal standard/surrogate solution to each calibration standard. A constant volume of methanol is also present in each standard. Analyze each calibration standard, beginning with the lowest and proceeding to the highest, and plot a calibration curve of peak heights vs. concentrations of the standards. Use an unweighted regression model that does not force the line through the origin, and calculate the coefficient of determination (COD), r^2 . An acceptable COD is 0.990 or higher. Alternatively, if the %RSD is less than or equal to 20%, the average calibration factor may be used.

Note: When preparing standards for the low-level soil closed system purge and trap method, all standards must contain the sodium bisulfate preservative, rather than reagent water.

14.1.3 The curve levels may vary due to the type and sensitivity of the detector(s) used. The standard levels used are 1, 4, 8, 12, 20, 40, 80 ug/L. Other levels may be substituted if sensitivity changes. However, the low point of the curve must be at or below the reporting limit.

14.1.4 For aqueous samples and methanol extracts, prepare the calibration standards in 50 mL volumetric flasks. Add the appropriate amount of intermediate stock standards (Section 12.4.1) as determined in 12.5.1, along with additional methanol to add up to a total solvent volume of 1.0 mL, to a 50 mL volumetric flask partially filled with water. Dilute to volume with water, cover with parafilm, and invert three times to mix. Pour off the contents in the neck of the volumetric, and fill a pre-cleaned 40 mL vial with the remainder. The autosamplers will add the IS/SS (Section 12.4.2) automatically before purging.

14.1.5 For low-level soil samples, prepare the calibration standards in a 5 mL Luerlock syringe. Add the appropriate amount of intermediate stock standards (Section 12.4.1) as determined in 12.5.1, along with additional methanol to add up to a total solvent volume of 100 μ L, to 5 mL of reagent water. Invert the syringe three times, then transfer to a new 40 mL vial.

Note: If the samples to be analyzed are preserved in sodium bisulfate, the curve must be prepared with the sodium bisulfate reagent (1g/5mL of water), rather than with reagent water.

- 14.1.6 Prepare a separate-source LCS standard (Section 12.4.6) to validate the calibration standards. The LCS must include all compounds being analyzed. Prepare and analyze the LCS as in Section 14.1.4 or 14.1.5 at a mid-range concentration. If all parameters are within 75 - 125% recovery, sample analysis may begin. If any of the recoveries are outside these limits, the analyst must repeat the test for the parameters that failed. If the second test fails, the problem must be corrected and a new calibration curve analyzed if necessary, and the process repeated. An LCS is analyzed following every initial calibration curve.
- 14.1.7 When analyzing soil/sludge samples by the low concentration method, a heated purge must be used during calibration. When analyzing soil/sludge samples by the high concentration method, the samples may be analyzed as a water sample, using an ambient purge temperature. Separate curves are required for heated and non-heated analyses.
- 14.2 Continuing calibration
- 14.2.1 The initial calibration must be verified each working day by the analysis of a continuing calibration standard (CCV). The CCV will be a mid-level standard containing all analytes of interest, and will be run after every 10 injections and after the final sample in the analytical sequence. The recovery of the analytes in the CCV must be within 85 - 115% of the true value. If these criteria are not met, all samples before and after the failing CCV will need to be reanalyzed. If the recovery of any analyte is greater than 115% but there are no positive results for that compound, samples do not require reanalysis.
- 14.2.2 As with the initial calibration, if low-level samples are analyzed containing sodium bisulfate, the CCV must also contain the sodium bisulfate preservative.
- 15.0 ANALYTICAL PROCEDURE**
- 15.1 Sample Analysis
- 15.1.1 Before initial use, a Vocab 3000 trap should be conditioned for at least one hour by baking at 260° C by using the helium purge gas. If other trapping materials are substituted for the Vocab 3000, follow the manufacturers recommendations for conditioning. Prior to daily use, the trap should also be baked out for 10 minutes at 260° C. After any period of inactivity, the GC column should be run through its temperature program, or ramped up to the final temperature and held for 15 minutes.
- 15.1.2 Set up the autosampler, concentrator, and GC as instructed in Section 9. For the soil samples, make sure the autosampler is set to inject an additional 5 mL of water to all standards and samples.
- 15.1.3 Analyze an instrument/method blank consisting of reagent water, methanol and internal/surrogate standard to check for contamination. If the concentration is less than the reporting limit for each analyte proceed to Step 15.1.4.
- 15.1.4 Analyze the seven initial calibration standards starting with the lowest and analyze sequentially in increasing order of concentration. Plot the calibration curve. The

coefficient of determination (r^2) must be ≥ 0.990 or the %RSD of the calibration factors must be $\leq 20\%$ in order to proceed.

- 15.1.5 Analyze 10 samples or less.
- 15.1.6 Analyze a CCV standard. The predicted response must not vary by more than $\pm 15\%$ or a new curve must be prepared.
- 15.2 After the GC run, identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with established retention time windows; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. If any questions remain, spike the sample or confirm by alternate column or GC/MS analysis.
- 15.2.1 Retention Time Windows
- 15.2.1.1 Make three injections of all target analytes over at least a 72-hour period. Calculate the standard deviation of the retention time of the three injections. For multi-response analytes, choose one major peak. The retention time window is calculated by multiplying the standard deviation by 3. For a positive identification, the analyte must elute within the absolute retention time of the ICV \pm the retention time window. All samples, quality control samples, and standards run after the ICV must have the analyte pass the retention time window criteria. Any samples run after a standard that fails the retention time window criteria must be re-analyzed.
- 15.2.1.2 Retention time windows must be re-run whenever a new column is installed, or after any major system maintenance is performed.
- 15.2.2 Analyte Confirmation
- 15.2.2.1 Positive results for analytes detected by this procedure will require some sort of confirmation. The following rules will apply regarding confirmation techniques.
- 15.2.2.2 When analyzing samples using the PID and ELCD in series, detection on both detectors will constitute as confirmation.
- 15.2.2.3 Samples or sites that have previously been analyzed by TriMatrix with positive results will not require confirmation. In addition, if historical data from a site is given to TriMatrix, and the results from the analysis match the historical results, no confirmation is required.
- 15.2.2.4 For samples with no historical data, all reportable results will be confirmed by GC/MS.
- 15.3 If the response for any compound exceeds the working range of the calibration curve, prepare a dilution of the sample.
- 15.3.1 Water Samples: Prepare a dilution of the sample from the second vial (or an aliquot in the previously saved 20 mL vial) in a volumetric flask. Total volume purged must be 5 mL. Organic free water must be used for the dilution.

15.3.2 Soil/Sludge/Waste Samples: Prepare a 1 g sample (13.2.1.1) if the expected concentration will be within linear range. If a larger dilution is needed prepare as in 13.2.2 or per EPA method 5035 as specified in TriMatrix SOP GR-04-105.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Aqueous Samples:

16.1.1 Concentration of analyte is determined as follows:

$$\text{Concentration in } \frac{\text{ug}}{\text{L}} = \text{CC} \times \text{DF}$$

Where:

CC = value obtained from the calibration curve (ug/L)

DF = dilution factor

16.1.2 Surrogate Recovery:

$$\% \text{ Recovery} = \left(\frac{\text{CC}}{\text{TV}} \right) \times 100$$

Where:

CC = value obtained from the calibration curve (ug/L)

TV = true value (50)

16.1.3 Matrix Spike Recovery:

$$\% \text{ Recovery} = \left(\frac{\text{CS} - \text{CC}}{\text{TV}} \right) \times 100$$

Where:

CS = Concentration of sample plus spike (ug/L)

CC = Concentration of sample (ug/L)

TV = True Value (40 or 60)

16.1.4 Relative Percent Difference of MS/MSD

$$\% RPD = \left(\frac{C_{MS} - C_{MSD}}{\left(\frac{C_{MS} + C_{MSD}}{2} \right)} \right) \times 100$$

C_{MS} = Concentration of matrix spike as calculated in 16.1.1.

C_{MSD} = Concentration of matrix spike duplicate as calculated in 16.1.

16.2 Soil/Sludge/Waste Samples (Low Concentration Method):

16.2.1 Concentration of analyte is determined as follows:

$$\frac{mg}{kg} = \left(\frac{CC \times 5 \text{ mL}}{W \times \% S} \right) \times \left(\frac{1L}{1000 \text{ mL}} \right) \times \left(\frac{1 \text{ mg}}{1000 \text{ } \mu\text{g}} \right) \times \left(\frac{1000 \text{ g}}{1 \text{ kg}} \right)$$

Where:

CC = value obtained from calibration curve (ug/L)

W = wet weight of sample (g)

% S = percent solids in decimal form (i.e., .90 = 90% solid). Used to calculate dry weight results for soils and sludges only. Wastes are calculated on a wet weight basis. See modified EPA Method 160.3.

16.2.2 Surrogate Recovery

$$\% \text{ Recovery} = \left(\frac{CS}{TV} \right) \times 100$$

Where:

CC = value obtained from the calibration curve (ug/L)

TV = true value (50)

16.2.3 Matrix Spike Recovery:

$$\% \text{ Recovery} = \left(\frac{CS - CC}{TV} \right) \times 100$$

Where:

CS = Concentration of sample plus spike (mg/kg) as calculated in 16.2.1

CC = Concentration of sample (mg/kg) as calculated in 16.2.1

TV = true spike value corrected for percent solids (0.040 or 0.06 mg/kg for 5 g samples and 0.200 or 0.300 mg/kg for 1 g samples prior to percent solids correction)

16.2.4 Relative Percent Difference of MS/MSD

$$\% RPD = \left(\frac{C_{MS} - C_{MSD}}{\left(\frac{C_{MS} + C_{MSD}}{2} \right)} \right) \times 100$$

Where:

C_{MS} = Concentration of matrix spike as calculated in 16.2.3.

C_{MSD} = Concentration of matrix spike duplicate as calculated in 16.2.3.

16.3 Soil/Sludge/Waste Samples (High Concentration Method)

16.3.1 Concentration of analyte is determined as follows:

$$\text{mg/kg} = \left(\frac{CC \times DF \times V}{W \times \% S} \right) \times \left(\frac{1L}{1000 \text{ ml}} \right) \times \left(\frac{1 \text{ mg}}{1000 \text{ ug}} \right) \times \left(\frac{1000 \text{ g}}{1 \text{ kg}} \right)$$

Where:

CC = value obtained from the calibration curve (ug/L)

DF = dilution factor

V = final volume of solvent added to sample during extraction or dilution (mL)

W = wet weight of sample extracted or diluted (g)

%S = percent solids in decimal form (i.e., 0.90 = 90% solid). Used to calculate dry weight results for soils and sludges only. Wastes are calculated on a wet weight basis. See Modified EPA Method 160.3.

16.3.2 Surrogate Recovery

$$\% \text{ Recovery} = \left(\frac{CC}{TV} \right) \times 100$$

CC = value obtained from the calibration curve (ug/L)

DF = dilution factor

TV = true value of surrogate spike

16.3.3 Matrix Spike Recovery:

$$\% \text{ Recovery} = \left(\frac{CS - CC}{TV} \right) \times 100$$

CS = Concentration of sample plus spike (mg/kg) as calculated in 16.3.1.

CC = Concentration of sample (mg/kg) as calculated in 16.3.1.
 TV = true spike value corrected for percent solids.

16.3.4 Relative Percent Difference of MS/MSD:

$$\% RPD = \left(\frac{C_{MS} - C_{MSD}}{\left(\frac{C_{MS} + C_{MSD}}{2} \right)} \right) \times 100$$

C_{MS} = Concentration of matrix spike as calculated in 16.3.1.
 C_{MSD} = Concentration of matrix spike duplicate as calculated in 16.3.1.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible not only for data quality but also for filling in all documentation correctly. It is essential to perform such tasks to provide the client with full defensible data.

17.2 LIMS Reporting

17.2.1 When the analyst has finished running a set of samples, data must be entered into the Element™ data system. First, samples are put into a bath of up to 20 samples. Samples of the same matrix and analysis method may be batched together. Next a sequence is created for all samples analyzed in a given 12-hour shift. When creating batches and sequences, it is imperative to enter all necessary information completely and correctly. This includes standards identification, spiking volumes, preparation dates and any other relevant information.

17.2.2 Data must be uploaded to Element™ in accordance with TriMatrix SOP GR-10-123.

17.2.3 Quality control recoveries are automatically calculated with out-of-control quality results being highlighted in red. Out-of-control data must be addressed in accordance with TriMatrix SOP GR-03-124.

17.2.4 method preparation blank (BLK) and a blank spike (LFB/BS) must be entered for each analysis sequence. Samples are associated with BLK and BS data by preparation date.

17.2.5 If internal chain-of-custody is required it is very important the COC for be filled in correctly and archived to the raw data folder. All sample possessions must be accounted for on the COC form.

17.3 Laboratory Required Paperwork

17.3.1 All run, maintenance, tape, and standard logbooks must be filled in completely and correctly. Corrections are to be made with a single lineout and then initialed and dated,

not a writeover, and blank lines in the run logbook should be Z'd out, initialed and dated.

17.3.2 All ICV and CCV standard runs must be archived in their correct binders.

17.3.3 All raw data (except for blanks, ICV runs and CCV runs) must be placed in the correct folder then given to the area supervisor for review and approval.

18.0 QUALITY ASSURANCE

18.1 Follow the procedures detailed in section 14.1 including daily method blank, calibration curve, and laboratory control sample analysis.

18.2 Continuing Calibration Verification (CCV)

18.2.1 The CCV consists of a mid-level standard prepared as in 14.1.4 or 14.1.5, which must be analyzed after every 10 samples and at the end of the analytical sequence. The recoveries of the CCV must fall within an 85-115% recovery range. If results fall outside this range, prior to the analysis of any samples, a second CCV may be immediately analyzed for each failed analyte. If results are still unacceptable, correct the problem and rerun all samples analyzed since the last acceptable CCV.

18.3 Method Preparation Blank (MPB)

18.3.1 An MPB must be analyzed each day prior to running samples, and must have no positive results above the laboratory reporting limits for any analyte. If this requirement is not met, sample analysis may not begin, and corrective action must be performed. The only exception to this is for common laboratory contaminants (i.e. methylene chloride), which may contain up to five times the laboratory reporting limit. However, any positive results reported for those analytes with concentrations between one and five times that reported in the blank will be qualified as estimated. If the samples to be analyzed are from methanol preparation, the MPB must contain 1 mL of methanol. If samples containing the bisulfate preservative are being analyzed, a MPB containing sodium bisulfate must also be analyzed.

18.4 Laboratory Fortified Blank (LFB)

18.4.1 A LFB is required with each 12-hour analytical batch, or every 20 samples, whichever is more frequent. It is taken from the results of the ICV at a mid-level concentration. The LFB serves as a check of method/extraction performance in the event that matrix spike recoveries are not within applicable control limits. Recoveries must be calculated and compared to LIMS control limits. Analysis must be stopped and the problem corrected if recoveries are outside the control limits. For aqueous and low-level soil samples, the LFB may be either from the same source or separate source as the curve. For high level soils and wastes, refer to Section 13.2.2.4 or TriMatrix SOP GR-04-105 (Method 5035) for LFB preparations. Any sample analyzed in a batch with a failing LFB must be re-analyzed for the failing parameters. If this is not possible, all data must be qualified as estimated.

18.5 Matrix Spikes (SPK, MSD)

- 18.5.1 Duplicate matrix spikes at a mid-level concentration should be analyzed for each batch of 20 samples of the same matrix. Recoveries and percent RPD are compared to LIMS control limits. Recoveries and/or percent RPD outside these limits must be qualified (see TriMatrix Volatile Laboratory Corrective Actions SOP - GR-03-104). See Section 12.0 for preparation procedures.
- 18.6 For both LFB and Matrix Spikes, a list of 5 compounds are reported as follows for method 8021B.
- 1,1-Dichloroethylene
 - Trichloroethylene
 - Chlorobenzene
 - Benzene
 - Toluene
- 18.7 Surrogate Recoveries
- 18.7.1 All surrogates must fall within LIMS control limits. If any surrogate compound is out of control due to obvious matrix problems, the sample will not be re-run and the sample will be qualified as estimated. If the sample has a surrogate that is out for no apparent reason, the sample will be re-run. If the re-run works, the first run will be discarded, and the second run reported. If many samples are out of control for no apparent reason, the instrument needs to be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re analysis of samples analyzed while the system was malfunctioning are necessary.
- 18.7.2 If a high level sample is not extracted or diluted and the true value of the surrogate is less than 5 ug/l, surrogate recoveries will not be reported.
- 18.8 Method 601/602 Modifications
- 18.8.1 This procedure was written primarily for method 8021B. However the following modifications are allowed for Method 601/602.
- 18.8.1.1 A 3 point curve can be used in place of a 5-point curve (Section 14.1.2).
 - 18.8.1.2 The recoveries of the CCV (Section 18.2) must fall between 80 - 120% of the true value.
 - 18.8.1.3 Every compound of interest that is on the Method 601/602 list must be in the matrix spikes and LFB; not just the limited list from Section 18.6.
 - 18.8.1.4 The MS/MSD frequency requirement is every 10 samples.
- 19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION**
- 19.1 Before the analysis 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.

- 19.2 Prepare four blank spikes in water at 20 ug/L. The spiking solution must be from a source other than that used to calibrate the instrument. Analyze as samples by going through every step in the procedure.
- 19.3 Input results to the IDC spreadsheet located on the laboratory intranet library to calculate percent recovery and relative standard deviation for each spiked analyte.
- 19.3.1 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 prepare waste samples by this procedure.
- 19.3.2 If any analyte fails any acceptance limit, locate and correct the problem then repeat the ENTIRE study successfully for the failed analyte. Replacing one apparent outlier result in the study is not permitted.
- 19.4 Repeated failure indicates a problem with the procedure and/or techniques used. If this occurs, locate the problem, correct the procedure and/or techniques used and repeat the IDC study successfully.
- 19.5 Samples may not be prepared by the analyst until a successful IDC study has been completed.
- 16.6 A successful continuing demonstration of capability (CDC) study must be completed annually by all analysts, by one of the following approaches:
- 19.6.1 By repeating the IDC study.
- 19.6.2 By exclusively preparing and analyzing an acceptable blind performance testing sample.
- 19.6.3 By exclusively preparing a method detection limit (MDL) study. The last four results of the study may be used.
- 19.7 A Method Detection Limit (MDL) study must be performed annually for both extraction procedures 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**
-

SOP Name: Volatile Organic Compounds
EPA Methods 601 and 602, SW-846 Method 8021B
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Date Revised: 2/17/12
Date Initiated: 11/27/95

- 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 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, December 1996, Method 8021B, "Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors", Revision 2, December, 1996*
- 22.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996, Method 5030B, "Purge-and-Trap for Aqueous Samples", Revision 2, December, 1996*
- 22.3 *Code of Federal Regulations, Title 40 Protection of Environment, Volume 19, July 1, 2001 Edition, Chapter I Environmental Protection Agency, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Purgeable Halocarbons, Method 601*
- 22.4 *Code of Federal Regulations, Title 40 Protection of Environment, Volume 19, July 1, 2001 Edition, Chapter I Environmental Protection Agency, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Purgeable Aromatics, Method 602*

23.0 ATTACHMENTS

- 23.1 Table 1 - Compound List and Reporting Detection Limits
- 23.2 Table 2 - Elution Order on DB-624
- 23.3 Table 3 - Elution Order on DB-VRX
- 23.4 Figure 1 - Example Chromatogram on DB-624 (HECD detector) Standard List
- 23.5 Figure 2 - Example Chromatogram on DB-624 (PID detector) Standard List
- 23.6 Figure 3 - Example Chromatogram on DB-624 (PID detector) Limited Aromatics List
- 23.7 Figure 4 - Example Chromatogram on DB-VRX (HECD detector) Standard List
- 23.8 Figure 5 - Example Chromatogram on DB-VRX (PID detector) Standard List

**Table 1
 Compound List And Reporting Limits**

<u>Analyte</u>	<u>CAS No.</u>	<u>Reporting Limits</u>	
		<u>Aqueous (ug/L)</u>	<u>Low Soil/High Soil (ug/kg)</u>
Benzene**	71-43-2	1.0	0.010/0.050
Bromobenzene	108-86-1	1.0	0.010/0.050
Bromochloromethane	74-97-5	1.0	0.010/0.050
Bromodichloromethane*	75-27-4	1.0	0.010/0.050
Bromoform*	75-25-2	1.0	0.010/0.050
Bromomethane*	74-83-9	1.0	0.010/0.050
n-Butylbenzene	104-51-8	1.0	0.010/0.050
sec-Butylbenzene	135-98-8	1.0	0.010/0.050
tert-Butylbenzene	98-06-6	1.0	0.010/0.050
Carbon tetrachloride*	56-23-5	1.0	0.010/0.050
Chlorobenzene*, **	108-90-7	1.0	0.010/0.050
Chloroethane*	75-00-3	1.0	0.010/0.050
2-Chloroethyl vinyl ether*	110-75-8	10	0.10/0.50
Chloroform*	67-66-3	1.0	0.010/0.050
Chloromethane*	74-87-3	1.0	0.010/0.050
2-Chlorotoluene	95-49-8	1.0	0.010/0.050
4-Chlorotoluene	106-43-4	1.0	0.010/0.050
Dibromochloromethane*	124-48-1	1.0	0.010/0.050
1,2-Dibromo-3-chloropropane	96-12-8	1.0	0.010/0.050
1,2-Dibromoethane	106-93-4	1.0	0.010/0.050
Dibromomethane	74-95-3	1.0	0.010/0.050
1,2-Dichlorobenzene*, **	95-50-1	1.0	0.010/0.050
1,3-Dichlorobenzene*, **	541-73-1	1.0	0.010/0.050

*Reported for 601
 **Reported for 602

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Table 1
Compound List And Reporting Limits
(Continued)

<u>Analyte</u>	<u>CAS No.</u>	<u>Reporting Limits</u>	
		<u>Aqueous</u> <u>(ug/L)</u>	<u>Low Soil/High Soil</u> <u>(mg/kg)</u>
1,4-Dichlorobenzene*, **	106-46-7	1.0	0.010/0.050
Dichlorodifluoromethane*	75-71-8	1.0	0.010/0.050
1,1-Dichloroethane*	75-34-3	1.0	0.010/0.050
1,2-Dichloroethane*	107-06-2	1.0	0.010/0.050
1,1-Dichloroethylene*	75-35-4	1.0	0.010/0.050
cis-1,2-Dichloroethylene*	156-59-2	1.0	0.010/0.050
trans-1,2-Dichloroethylene*	156-60-5	1.0	0.010/0.050
1,2-Dichloropropane	78-87-5	1.0	0.010/0.050
1,3-Dichloropropane	142-28-9	1.0	0.010/0.050
2,2-Dichloropropane	594-20-7	1.0	0.010/0.050
1,1-Dichloropropylene	563-58-6	1.0	0.010/0.050
cis-1,3-Dichloropropylene*	10061-01-5	1.0	0.010/0.050
trans-1,3-Dichloropropylene*	10061-02-6	1.0	0.010/0.050
Ethylbenzene**	100-41-4	1.0	0.010/0.050
Hexachlorobutadiene	87-68-3	1.0	0.010/0.050
Isopropylbenzene	98-82-8	1.0	0.010/0.050
Isopropyltoluene, para	99-87-6	1.0	0.010/0.050
Methylene chloride*	75-09-2	1.0	0.010/0.050
Methyl(tert)butyl ether	1634-04-4	50	0.10/2.5
Naphthalene	91-20-3	1.0	0.010/0.050
n-Propylbenzene	103-65-1	1.0	0.010/0.050
Styrene	100-42-5	1.0	0.010/0.050
1,1,1,2-Tetrachloroethane	690-20-6	1.0	0.010/0.050

*Reported for 601.

**Reported for 602.

Table 1
Compound List And Reporting Limits
(Continued)

Analyte	CAS No.	Reporting Limits	
		Aqueous (ug/L)	Low Soil/High Soil (ug/kg)
1,1,2,2-Tetrachloroethane*	79-34-5	1.0	0.010/0.050
Tetrachloroethylene*	127-18-4	1.0	0.010/0.050
Toluene**	108-88-3	1.0	0.010/0.050
1,2,3-Trichlorobenzene	87-61-6	1.0	0.010/0.050
1,2,4-Trichlorobenzene	120-82-1	1.0	0.010/0.050
1,1,1-Trichloroethane*	71-55-6	1.0	0.010/0.050
1,1,2-Trichloroethane*	79-00-5	1.0	0.010/0.050
Trichloroethylene*	79-01-6	1.0	0.010/0.050
Trichlorofluoromethane*	75-69-4	1.0	0.010/0.050
1,2,3-Trichloropropane	96-18-4	1.0	0.010/0.050
1,2,4-Trimethylbenzene	95-63-6	1.0	0.010/0.050
1,3,5-Trimethylbenzene	108-67-8	1.0	0.010/0.050
Vinyl chloride*	75-01-4	1.0	0.010/0.050
Xylene, meta & para**	999-9-9***	2.0	0.020/0.100
Xylene, ortho**	95-47-6	1.0	0.010/0.050
Xylene, Total **	1330-20-7	3.0	0.030/0.150

*Reported for 601.
 **Reported for 602.
 ***m-xylene 108-38-3
 ***p-xylene 106-42-3

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Table 2
Elution Order & Detector Response – DB-624

<u>Parameter</u>	<u>ELCD Response</u>	<u>PID Response</u>
1. Dichlorodifluoromethane	X	---
2. Chloromethane	X	---
3. Vinyl chloride	X	X
4. Bromomethane	X	---
5. Chloroethane	X	---
6. Trichlorofluoromethane	X	---
7. 1,1-Dichloroethylene	X	X
8. Methylene chloride	X	---
9. trans-1,2-Dichloroethylene	X	X
10. Methyl(tert)butyl ether	---	X
11. 1,1-Dichloroethane	X	---
12. cis-1,2-Dichloroethylene	X	X
13. 2,2-Dichloropropane	X	---
14. Bromochloromethane	X	---
15. Chloroform	X	---
16. 1,1,1-Trichloroethane	X	---
17. Carbon tetrachloride	X	---
18. 1,1-Dichloropropylene	X	X
S1. 1,2-Dichloroethane-d4	X	---
19. 1,2-Dichloroethane	X	---
20. Benzene	---	X
11 Fluorobenzene (Internal)	---	X
21. Trichloroethylene	X	X
22. 1,2-Dichloropropane	X	---

*Depending on parameter list, may be used as a surrogate.

Note: Compounds in bold are usually analyzed for. If additional compounds are requested, a different temperature program may be needed (see section 9.2).

Table 2
Elution Order & Detector Response – DB-624
(Continued)

<u>Parameter</u>	<u>ELCD Response</u>	<u>PID Response</u>
23. Dibromomethane	X	---
24. Bromodichloromethane	X	---
25. 2-Chloroethyl vinyl ether	X	X
S2 α,α,α -Trifluorotoluene (surrogate)	---	X
26. cis-1,3-Dichloropropylene	X	X
27. Toluene	---	X
28. trans-1,3-Dichloropropylene	X	X
29. 1,1,2-Trichloroethane	X	---
30. Tetrachloroethylene*	X	X
12. 2-Bromo-3-Chloropropane	X	---
31. 1,3-Dichloropropane	X	---
32. Dibromochloromethane	X	---
33. 1,2-Dibromoethane	X	---
34. Chlorobenzene	X	X
35. 1,1,1,2-Tetrachloroethane	X	---
36. Ethylbenzene	---	X
37. Xylene, meta & para	---	X
38. Xylene, ortho	---	X
39. Styrene	---	X
40. Bromoform	X	---
41. Isopropylbenzene	---	X
42. 1,1,2,2-Tetrachloroethane	X	---

*Depending on parameter list, may be used as a surrogate.

Note: Compounds in bold are those usually analyzed for. If additional compounds are requested, a different temperature program may be needed (see section 9.2).

Table 2
Elution Order & Detector Response – DB-624
(Continued)

<u>Parameter</u>	<u>ELCD Response</u>	<u>PID Response</u>
43. 1,2,3-Trichloropropane	X	---
44. Bromobenzene	X	X
45. n-Propylbenzene	---	X
46. 2-Chlorotoluene	X	X
47. 4-Chlorotoluene*	X	X
48. 1,3,5-Trimethylbenzene	---	X
49. tert-Butylbenzene	---	X
50. 1,2,4-Trimethylbenzene	---	X
51. sec-Butylbenzene	---	X
52. Isopropyltoluene, para	---	X
53. 1,3-Dichlorobenzene	X	X
54. 1,4-Dichlorobenzene	X	X
55. n-Butylbenzene	---	X
56. 1,2-Dichlorobenzene	X	X
57. 1,2-Dibromo-3-chloropropane	X	---
58. 1,2,4-Trichlorobenzene	X	X
59. Hexachlorobutadiene	X	X
60. Naphthalene	---	X
61. 1,2,3-Trichlorobenzene	X	X

*Depending on parameter list, may be used as a surrogate.

Note: Compounds in bold are those usually analyzed for. If additional compounds are requested, a different temperature program may be needed (see section 9.2).

Table 3
Elution Order On DB-VRX – DB-VRX

<u>Parameter</u>	<u>ELCD Response</u>	<u>PID Response</u>
1. Dichlorodifluoromethane	X	---
2. Chloromethane	X	---
3. Vinyl chloride	X	X
4. Bromomethane	X	---
5. Chloroethane	X	---
6. Trichlorofluoromethane	X	---
7. 1,1-Dichloroethylene	X	X
8. Methylene chloride	X	---
9. trans-1,2-Dichloroethylene	X	X
10. Methyl(tert)butyl ether	---	X
11. 1,1-Dichloroethane	X	---
12. cis-1,2-Dichloroethylene	X	X
13. Bromochloromethane	X	---
14. Chloroform	X	---
15. 2,2-Dichloropropane	X	---
S1 1,2-Dichloroethane-d4	X	---
17. 1,2-Dichloroethane	X	---
18. 1,1,1-Trichloroethane	X	---
19. 1,1-Dichloropropylene	X	X
20. Carbon tetrachloride	X	---
21. Benzene	---	X
11 Fluorobenzene (Internal)	---	X
22. Dibromomethane	X	---
23. 1,2-Dichloropropane	X	---
24. Trichloroethylene	X	X

*Depending on parameter list may be used as a surrogate.

Note: Compounds in bold are those usually analyzed for. If additional compounds are requested, a different temperature program may be needed (see section 9.2).

Table 3
Elution Order On DB-VRX – DB-VRX
(Continued)

<u>Parameter</u>	<u>ELCD Response</u>	<u>PID Response</u>
25. Bromodichloromethane	X	---
S2 α,α,α -Trifluorotoluene (surrogate)	---	X
26. 2-Chloroethyl vinyl ether	X	X
27. cis-1,3-Dichloropropylene	X	X
28. trans-1,3-Dichloropropylene	X	X
29. 1,1,2-Trichloroethane	X	---
12. 2-Bromo-3-Chloropropane (Internal)		X
30. Toluene	---	X
31. 1,3-Dichloropropane	X	
32. Dibromochloromethane	X	---
33. 1,2-Dibromoethane	X	---
34. Tetrachloroethylene*	X	X
35. 1,1,1,2-Tetrachloroethane	X	---
36. Chlorobenzene	X	X
37. Ethylbenzene	---	X
38. Xylene, meta & para	---	X
39. Bromoform	X	---
40. Styrene	---	X
41. 1,1,2,2-Tetrachloroethane	X	---
42. Xylene, ortho	---	X
43. 1,2,3-Trichloropropane	X	---
44. Isopropylbenzene	---	X

*Depending on parameter list, may be used as a surrogate.

Note: Compounds in bold are those usually analyzed for. If additional compounds are requested, a different temperature program may be needed (see section 9.2).

Table 3
Elution Order On DB-VRX – DB-VRX
(Continued)

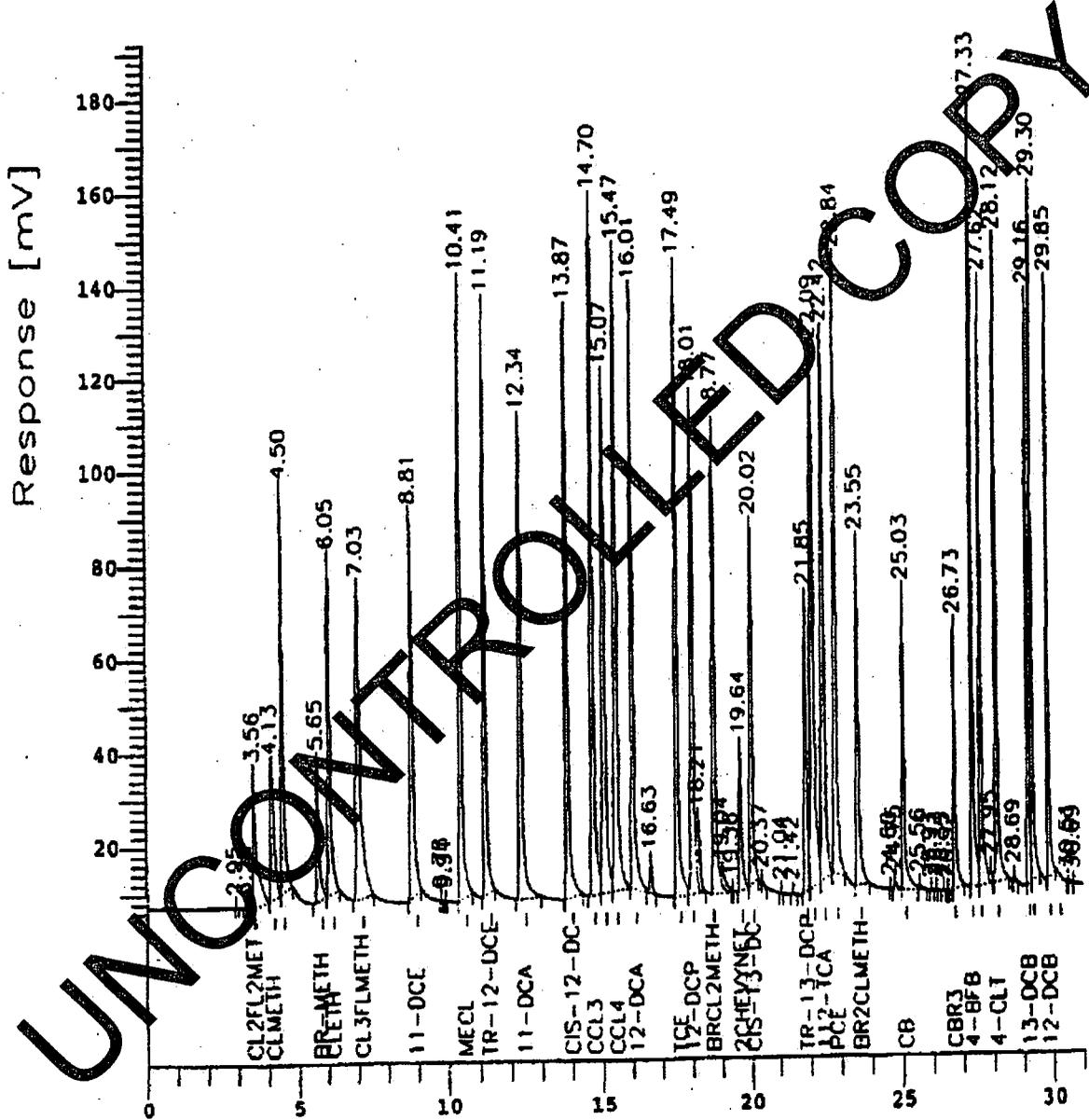
<u>Parameter</u>	<u>ELCD Response</u>	<u>PID Response</u>
45. Bromobenzene	X	X
46. n-Propylbenzene	---	X
47. 2-Chlorotoluene	X	X
48. 4-Chlorotoluene*	X	X
49. 1,3,5-Trimethylbenzene	---	X
50. tert-Butylbenzene	---	X
51. 1,2,4-Trimethylbenzene	---	X
52. sec-Butylbenzene	---	X
53. 1,3-Dichlorobenzene	X	X
54. 1,4-Dichlorobenzene	X	X
55. Isopropyltoluene, para	---	X
56. 1,2-Dichlorobenzene	X	X
57. n-Butylbenzene	---	X
58. 1,2-Dibromo-3-chloropropane	X	---
59. 1,2,4-Trichlorobenzene	X	X
60. Naphthalene	---	X
61. Hexachlorobutadiene	X	X
62. 1,2,3-Trichlorobenzene	X	X

*Depending on parameter list, may be used as a surrogate.

Note: Compounds in bold are those usually analyzed for. If additional compounds are requested, a different temperature program may be needed (see section 9.2).

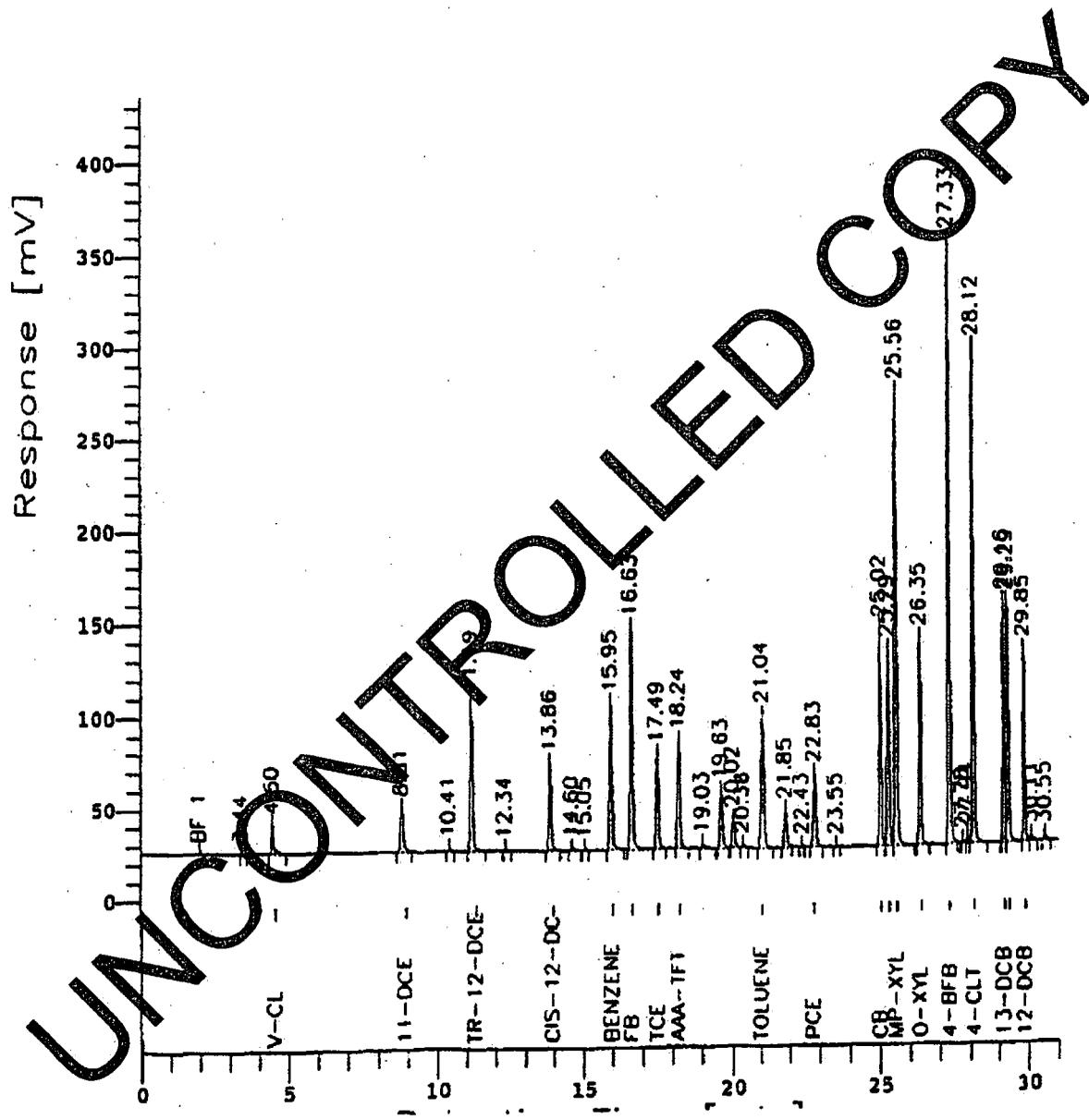
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Figure 1
 Sample Chromatograms
 Column: DB-624 75m .53 id capillary Detector: ELCD



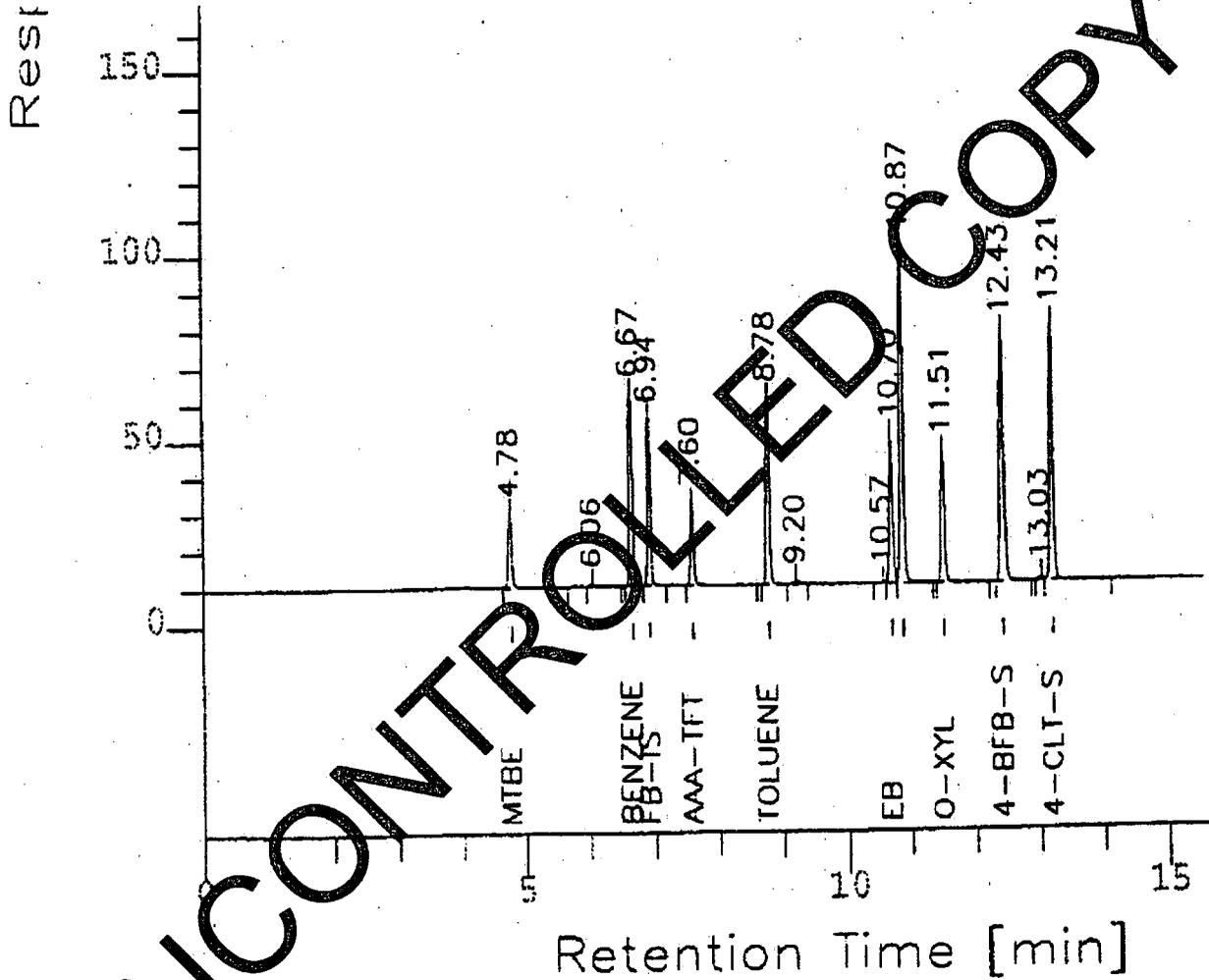
Note: See Attachment 23.2 - Table 2 for compound identification. Chromatogram shown is for the normal list of compounds (**BOLDFACE** compounds on Table 2).

Figure 2
Sample Chromatograms
Column: DB-624 75m .53 id capillary Detector: PID



Note: See Table 2 for compound identification. Chromatogram shown is for the normal list of compounds (**BOLDFACE** compounds on Table 2).

Figure 3
Sample Chromatograms
Column: DB-624 30m .53 id capillary Detector: PID



Note: See Table 2 for compound identification. Chromatogram shown is the normal aromatic list analyzed including 602 compounds and MTBE.

Figure 4
Sample Chromatograms
Column: DB-VRX 75m .53 id capillary Detector: ELCD

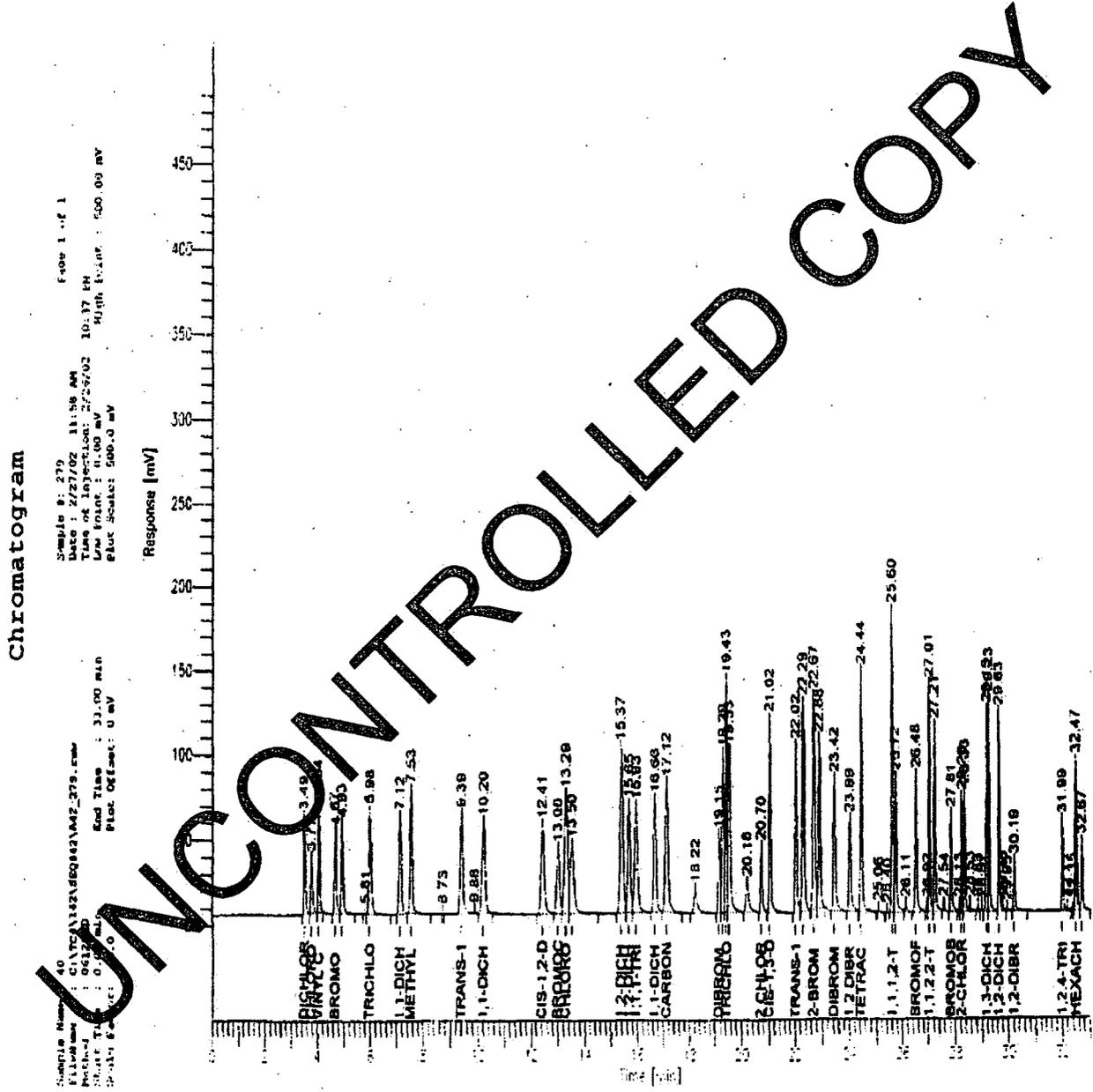
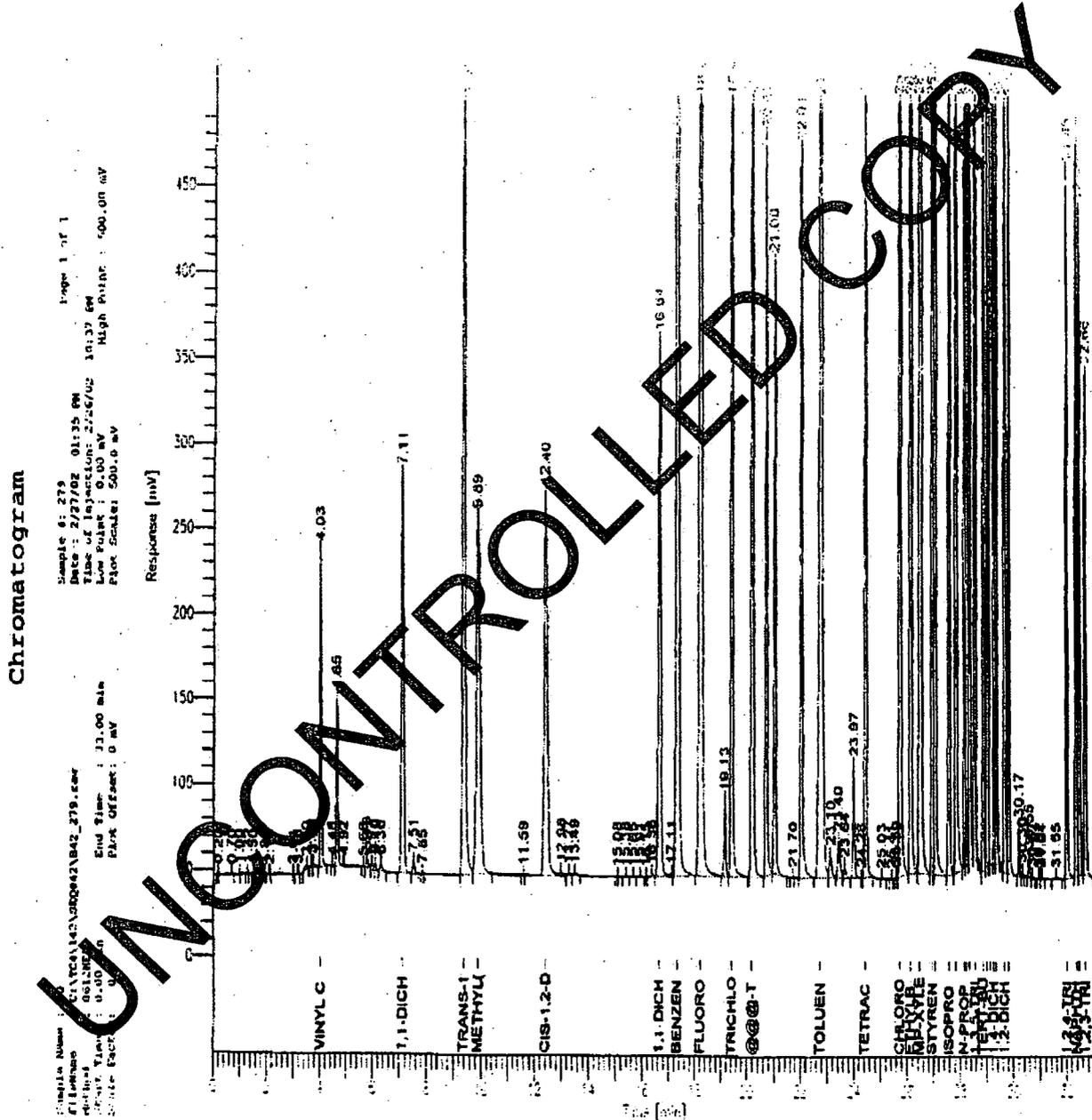


Figure 5
Sample Chromatograms
Column: DB-VRX 75m .53 id capillary Detector: PID





STANDARD OPERATING PROCEDURE

Organochlorine Pesticides Analysis by Gas Chromatography (GC)

EPA Methods 608 and 608.2
SW-846 Method 8081B

APPROVALS:

Area Supervisor: Jodi Blouw Date: 4-20-11
Jodi L. Blouw

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By: Daniel J. Mierendorf

Total Number of Pages: 31

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If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
10/4/12	Jodi Blouw	10/4/13

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 μ L.

4.0 PARAMETER OR COMPOUND LIST

- 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, *Florasil Column Cleanup*, Method 2620, 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, *Basic/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, Florasil 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 vendor.
- 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 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 $4 \pm 1^\circ\text{C}$, 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 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 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.
- 10.4 ECD Wipe Test and ⁶³Ni 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 $4 \pm 2^\circ \text{C}$ 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.
- 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:

$$\frac{\text{mg compound}}{0.100 \text{ L solvent}} = \text{mg/L (or ug/mL)}$$

For Example:

$$\frac{101 \text{ mg Mirex}}{0.100 \text{ L Hexane}} = 1010 \text{ mg/L Mirex}$$

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 solutions 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:

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 H_x = 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
 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:

$$CF = \frac{\text{Peak Height}}{\text{Analyte Concentration in ng/uL}}$$

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 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
- 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.

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):

$$\text{Concentration (mg/L)} = \frac{\text{Height Counts of the Analyte}}{\text{Average CF}}$$

- 14.3.1.1 For average CF, verify by percent difference between the average and continuing factors using the following equation:

$$\text{Percent Difference} = \frac{CF_v - CF_m}{CF_m} \times 100$$

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:

R₁ = Actual value
 R₂ = Quantitated value

14.3.2 Percent difference or percent drift must be within $\pm 20\%$ of the actual value for samples analyzed by method 8081B. Percent difference or percent drift must be within $\pm 15\%$ of the actual value for samples analyzed by method 608. Percent difference or percent drift must be within $\pm 10\%$ 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.

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.
- $$RPD = \frac{|R_1 - R_2|}{R_1 + R_2} \times 200$$
- 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 (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 than one 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:

$$\text{ng / uL (Average CF)} = \frac{\text{PeakHeight}(s)}{\text{CF}}$$

$$\text{ng / uL (Linear)} = \frac{v - b}{m}$$

a, b, c coefficients (nonlinear/Excel) = linest(y-values,x-values^{1,2},true)

- 16.3 Next, calculate sample concentration:

For water samples:

$$\text{sample concentration (ug / L)} = \frac{(\text{ng / uL})(V_e)(DF)}{V_s}$$

For soil samples:

$$\text{sample concentration (mg / kg)} = \frac{(\text{ng / uL})(V_e)(DF)(1000)}{V_s (PS)}$$

where:

- V_e = Total extract volume (μL)
- DF = Post-extraction dilution factor ($\mu\text{L} + \mu\text{L}$)/(total μL)
- V_s = 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 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.

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:

$$\% \text{ recovery} = \frac{(A_{spk} - A_{smp})}{SPK} \times 100$$

where:

A_{spk} = Actual concentration found in the spiked sample in ug/L

A_{smp} = Concentration found in non-spiked sample in ug/L

$$SPK = \left(\frac{\text{concentration of spiked standard in ug/L} \times \text{ml spiked}}{\text{initial sample volume in L}} \right)$$

18.4.4 The statistical duplication control limit is calculated and maintained in Element™. Analyte spike duplication is based on the following calculation:

$$\%RPD = \frac{|(SPK_1 - SPK_2)|}{\left[\frac{(SPK_1 + SPK_2)}{2}\right]} \times 100$$

where:

SPK₁ = ug/L found in matrix spike (MS)

SPK₂ = 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.
- 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).
- 18.4.8 If insufficient sample is received to perform a matrix spike/matrix spike duplicate, the report must be narrated as follows:
- GN006 – Due to insufficient sample volume received, no matrix QC is available with this sample batch.
- 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:
- $$RR_i(\%) = (100 \cdot R_i) / [(X_1 + X_2) / 2]$$
- Where:
- R_i = Absolute difference between the duplicate measurements X₁ and X₂ in ug/L
- X_i = Average concentration found [(X₁ + X₂)/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 $\mu\text{g/L}$ or mg/kg .

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 of the *Testing 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.
- 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
- 23.11 Figure 7 – Preparation Batch Detail Report Example
- 23.12 Figure 10 – Instrument Run Log Example

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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	50-29-2
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	16.87	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	15.71	15.28	5103-74-2
Endrin ketone	17.10	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
Kepon	15.32	15.34	143-50-0
Mex	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'-	23.46	27.84	-
Hexabromobiphenyl (BP-6)	18.01	21.74	-
Hexabromobenzene (HBB)	18.01	21.74	-
Tris-(2,3-dibromopropyl)	**	**	-
phosphate	**	**	-
Chlordane, Technical Grade	**	**	57-74-9
Toxaphene	**	**	8001-35-2

**Multi-response compound

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

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Table 3
Method Detection Limit Study Example

 SEMI-VOLATILE/VOLATILE/METALS LABORATORY
 INSTRUMENT NUMBER 158A 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
alpha BHC	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
gamma BHC	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
beta BHC	608/8081A	3/15/2004	0.008	ug/L	0.007	0.007	0.007	0.007	0.005	0.001	0.006	0.005	69%	0.0021	0.0067
delta BHC	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
Heptachlor	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
Aldrin	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
Heptachlor Epoxide	608/8081A	1/10/2004	0.008	ug/L	0.008	0.008	0.008	0.008	0.005	0.008	0.009	0.008	99%	0.0014	0.0043
g-Chlordane	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
a-Chlordane	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
4,4'-DDE	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
Endosulfan I	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
Dieldrin	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
Endrin	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
4,4'-DDD	608/8081A	1/10/2004	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
Endosulfan II	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
4,4'-DDT	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
Endrin Aldehyde	608/8081A	1/10/2004	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
methoxychlor	608/8081A	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
endosulfan sulfate	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
Endrin Ketone	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

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SOP Name: Organochlorine Pesticides Analysis by Gas Chromatography (GC)
 EPA Methods 608/608.2, SW-846 Method 8081B
 SOP Number: GR-03-120 page 24 of 31

Revision Number: 4.6
 Date Revised: 9/19/11
 Date Initiated: 10/12/94

Figure 1
 Analytical Standards Report Example

Analytical Standard Record
 TriMatrix Laboratories, Inc.
 7090175

Description:	pest L1	Expires:	Jan-02-08
Standard Type:	Calibration Star	Prepared By:	Sep-06-07 Daniel J. Mierendorf
Solvent:	Solvent Lot #	Department:	Sensivolutions GC
Final Volume (mL):	2	Last Edit:	Sep-06-07 16:42 by DJM
Vials:	1		

Analyte	CAS Number	Concentration	Units
alpha-Chlordane	5103-71-9	0.005	ug/mL
4,4'-DDD	72-34-8	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]	72-33-9	0.005	ug/mL
4,4'-DDE	72-33-9	0.005	ug/mL
4,4'-DDD [2C]	72-34-8	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]	92-43-5	0.005	ug/mL
Methoxychlor	92-43-5	0.005	ug/mL
Heptachlor Epoxide [2C]	324-57-3	0.005	ug/mL
Heptachlor Epoxide	324-57-3	0.005	ug/mL
Heptachlor [2C]	6-44-8	0.005	ug/mL
Heptachlor	6-44-8	0.005	ug/mL

Reviewed By: _____ Date: _____

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 Alderlactone [2C]	7421-93-4	0.005	ug/mL
Endrin Alderlactone	7421-93-4	0.005	ug/mL
Endrin	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. Mierendorf	Jan-02-08	Sep-06-07 16:42 by DJM	1

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) $r^2 = 0.997960$
 Calibration Curve = $(11057.842158) + (2099938.639920)X$



Level Name	Observed X-Value	Calculated 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	0.015000	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	0.150000	0.157904	0.002096	1.327	342646.215	347048.025	-4401.810	-1.268

Figure 3
Pesticide Chromatogram On RTX CLP Column

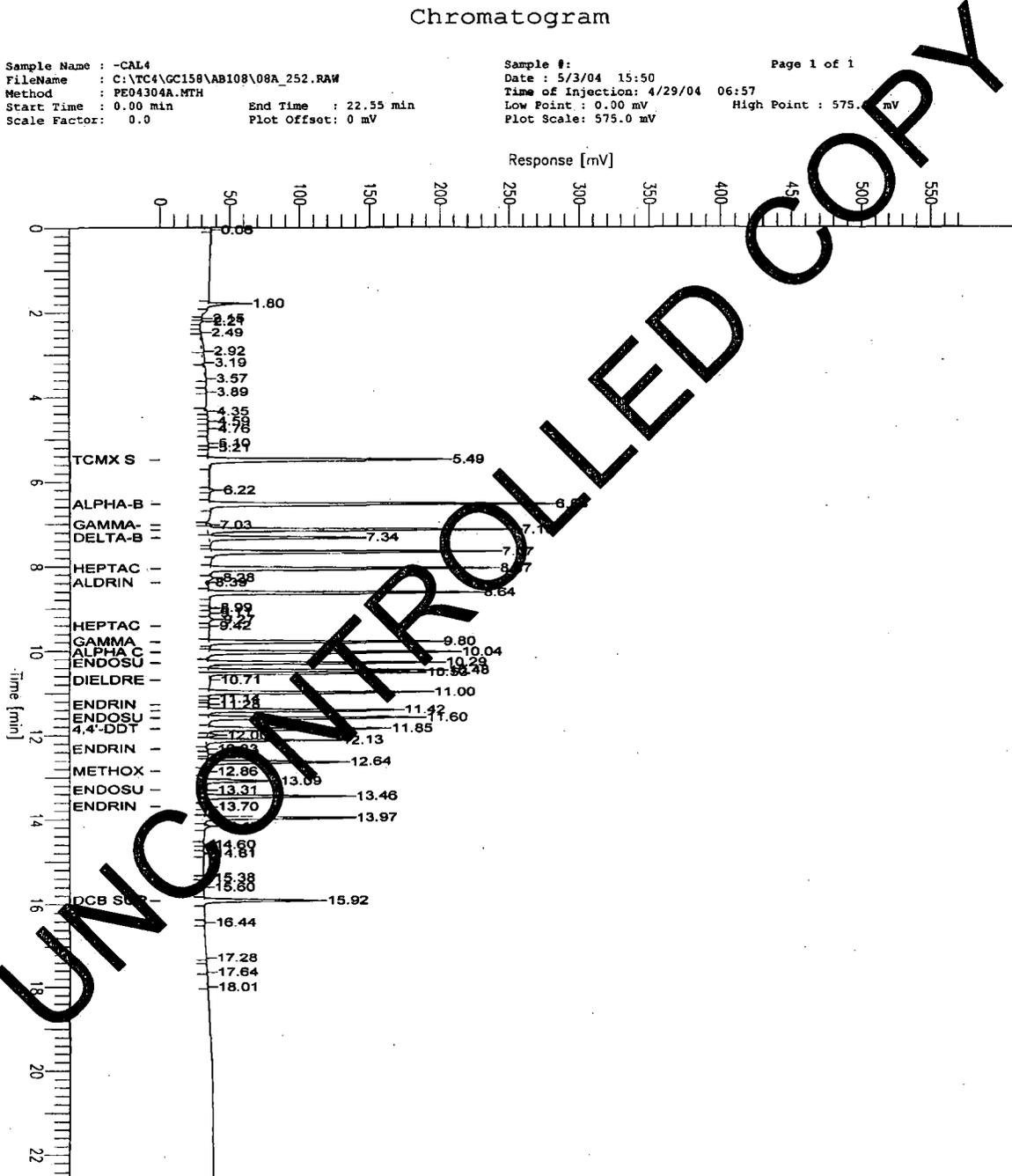


Figure 4
Pesticide Chromatogram on RTX CLP2 Column

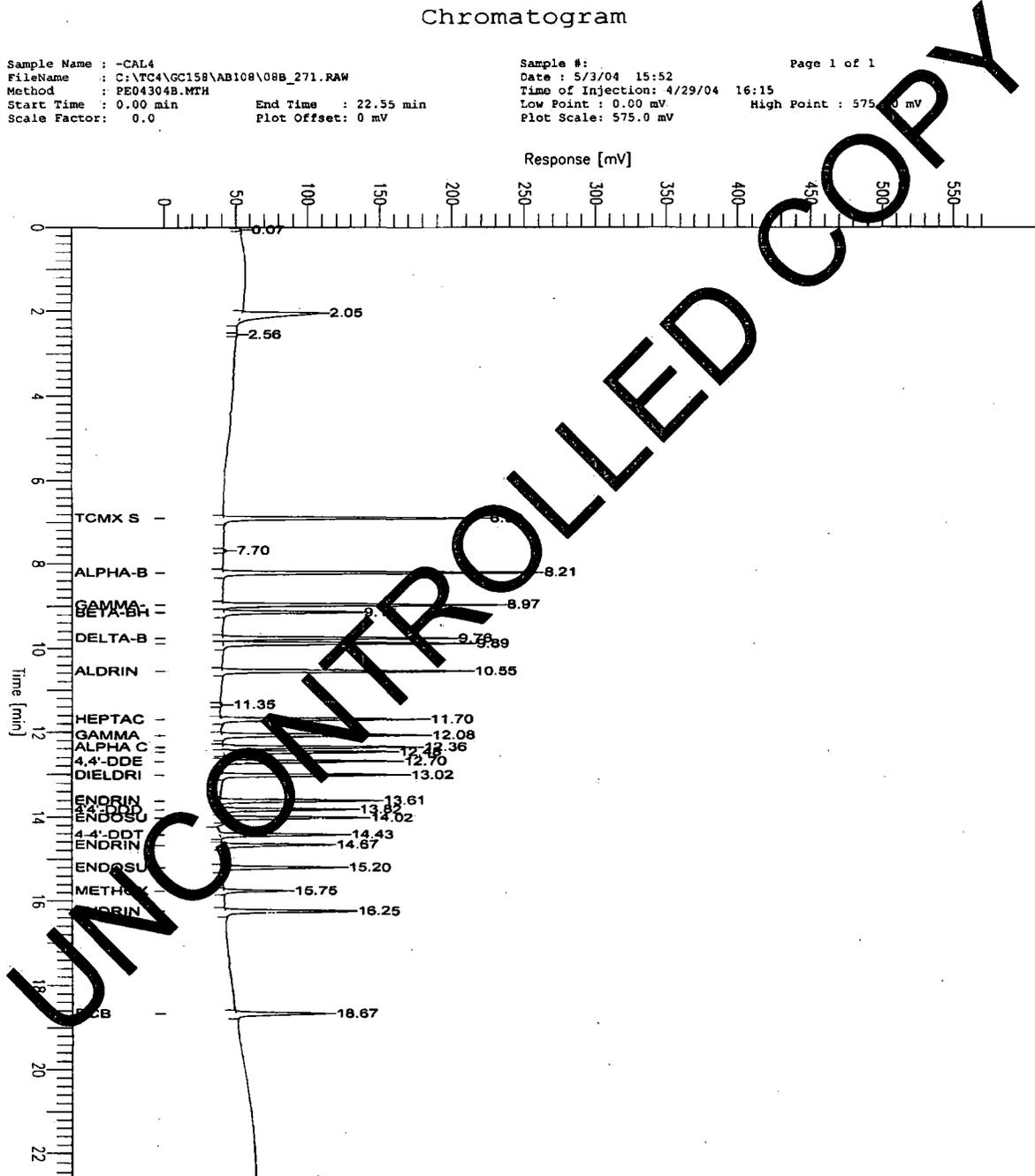


Figure 5
Chlordane Multi-Component Pesticide Chromatogram

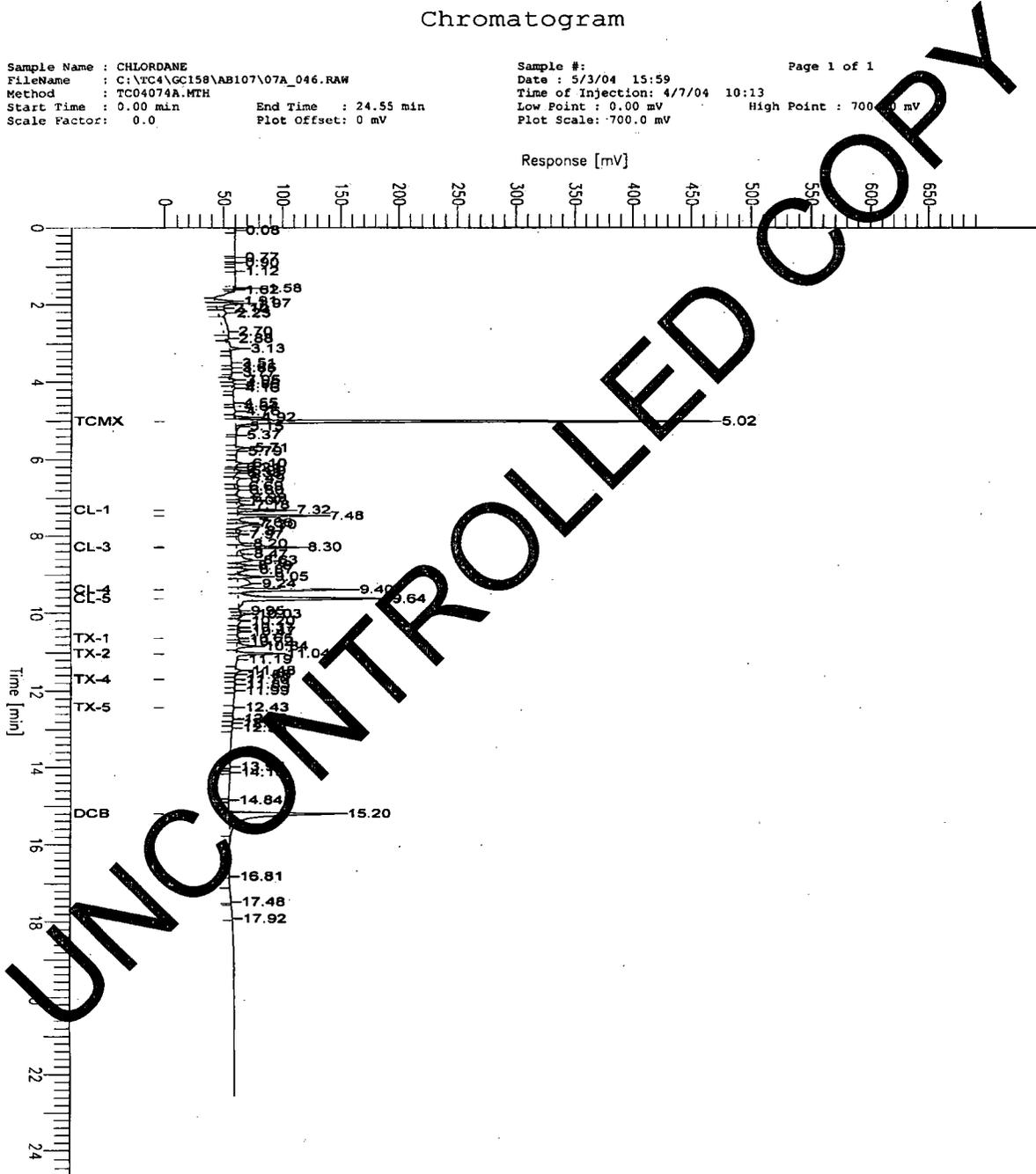


Figure 6
 Data Review Report Example

TriMatrix Laboratories, Inc.

Data Review Report [REDACTED]
 Sequence = 7102946 Page 1 of 21

on 12/21/2007 at 5:45

SampleID	Analyst	IResult	DIn	FResult	FMRI	Qualifier	Recovery	RPD	SampleID	Analyst	Result	
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)	0.0686	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	40	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	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	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



SOP Name: Organochlorine Pesticides Analysis by Gas Chromatography (GC)
 EPA Methods 608/608.2, SW-846 Method 8081B
 SOP Number: GR-03-120

Revision Number: 4.6
 Date Revised: 9/19/11
 Date Initiated: 10/12/94

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Figure 7
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH **0712971** Page 1 of 1
 Semivolatiles GC, Water, 3510C Liquid-Liquid Extraction
 Surrogate #1 = 7070019 (Pre-Prep)
 Batch Comments: (none)

Printed: 12/21/2008 5:50:09PM

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0710183	8081A PESTs (master list)	0710183	8081A MDEQ Pests	0710183	8081A MDEQ 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					Added for BatchQC in: 0712971
0710183-24	A	Oct-25-07 18:18	ASC	1000	2	1000					
0710183-24	A	Oct-25-07 18:18		1000	2	1000					Added for BatchQC in: 0712971
0710183-25	A	Oct-25-07 18:18	ASC	1000	2	1000					
0710183-25	A	Oct-25-07 18:18		1000	2	1000					Added for BatchQC in: 0712971
0710183-25	A	Oct-25-07 18:18		1000	2	1000					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		7070887	200	LCS	toxaphene
0710183-30	A	Oct-31-07 15:18	KB9	1000	2	1000					Added for BatchQC in: 0712971
0710183-30	A	Oct-31-07 15:18	KB9	1000	2	1000					Added for BatchQC in: 0712971
0710183-30	A	Oct-31-07 15:18	KB9	1000	2	1000					GOOD WP Total Chlordane
0710183-36	A	Oct-31-07 15:18	KB9	1000	2	1000					Added for BatchQC in: 0712971
0710183-36	A	Oct-31-07 15:18	KB9	1000	2	1000					GOOD WP Toxaphene
0710183-36	A	Oct-31-07 15:18	KB9	1000	2	1000					Added for BatchQC in: 0712971

Comments	Analyst Initials
----------	------------------

bch_TriMatrix.rpt

SOP Name: Organochlorine Pesticides Analysis by Gas Chromatography (GC)
 EPA Methods 608/608.2, SW-846 Method 8081B
 SOP Number: GR-03-120

Revision Number: 4.6
 Date Revised: 9/19/11
 Date Initiated: 10/12/94

Figure 8
 Instrument Run Log Example



Instrument 199 HP6890		Date: 1/20/08		Sequence #: AA66		Date Archived:		Analyt			
Instrument Settings / Injection Volume			GC Program			Quantitation Information / Breakdown			Routine Maintenance Items		
Column Type: STXCLP / STXCLP2	Initial:					Curve Date:			New Column Date:		
Injection Volume: 10L / 0.1L	Hold:					Curve Type: Regression or Average CF			Column Checked: 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	TV/CCV Check (85-115)	Sample Notes, Standard Numbers, Analytical Batch Information, etc.			
PEM	147	12:00	PEWJ08A.S		RC		PASS	7110868			
Inst BLK	148	12:35									
Tox ICL	149	13:11						8020776			
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							8010108			
P5(L)	156							8010193			
PEM	157	17:57						7110868			
080167-03	158	18:32									
↓ -01	159	19:08									
080157-03	160	19:44									
↓ -02	161	20:19									
↓ -01	162	20:55									



STANDARD OPERATING PROCEDURE

Polychlorinated Biphenyls (PCB) by Gas Chromatography

EPA Method 608
SW-846 Method 8082A

APPROVALS:

Area Supervisor: Jodi Blouw
Jodi L. Blouw

Date: 2-27-12

QA Officer: Tom Boocier
Tom Boocier

Date: 2-17-12

President: Douglas E. Kriscunas
Douglas E. Kriscunas

Date: 3-5-2012

Procedure Number: GR-03-128
Revision Number: 2.7

Date Initiated: 4/2/98
Effective Date: 2/28/12

Date Revised: 2/17/12
Pages Revised: All

By: Mark S. Zwiefka

Total Number of Pages: 34

UNCONTROLLED COPY

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
<u>12-17-12</u>	<u>Jodi Blouw</u>	<u>12-17-13</u>

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 accurate 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 This procedure uses a dual-column system in addition to characteristic pattern matching to confirm the identity of any Aroclor found.
- 1.3 Default reporting limits (RL) are listed in Attachment 23.1.

Note: Reporting limits are highly matrix dependent and the default reporting limit might not be achievable with all sample matrices.
- 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 This analysis is restricted for use by or under the supervision of analysts experienced in gas chromatography and chromatographic interpretation of PCBs.

2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, February, 2007, Method 8082A, "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 1.0 μ L.
- 3.2 After separation by the column, 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 SOP GR-04-101, *Semi-Volatile Organic Laboratory Corrective Actions*, latest revision
- 5.2 TriMatrix SOP GR-09-105, *Waste Dilution Preparation Method for PCBs and Pesticides*, latest revision
- 5.3 TriMatrix SOP GR-09-107, *Extraction of Organochlorine Pesticides and PCBs in Water*, latest revision
- 5.4 TriMatrix SOP GR-09-108, *Extraction of PCBs/Pesticides in Soil, Sludge, and Wastes*, latest revision
- 5.5 TriMatrix SOP GR-09-109, *Sulfur Cleanup*, latest revision
- 5.6 TriMatrix SOP GR-09-110, *Sulfuric Acid Cleanup*, latest revision
- 5.7 TriMatrix SOP GR-09-111, *Florisil Column Cleanup*, latest revision
- 5.8 TriMatrix SOP GR-09-120, *Florisil/Silica Gel cleanup of PCBs, Toxaphene, Chlordane*, latest revision
- 5.9 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision
- 5.10 TriMatrix 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 best 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 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 Wear a laboratory coat and approved safety glasses while in the laboratory. In addition, disposable gloves must be worn whenever samples or reagents are handled. The only exception to the safety glasses requirement policy is when sitting at a computer station.

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.

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 exposure 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 Waste samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-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.

7.7 PCBs can cause irritation upon contact and are carcinogenic. Open, prepare and dispense standards and solutions only in a fume hood.

7.8 The solvents used 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.

- 7.9 All analyzed sample extracts and expired standards must be disposed of properly. Consolidate and segregate all samples and extracts testing positive for any concentration of PCBs as PCB-containing waste.
- 7.10 If a PCB or solvent spill occurs, notify the area supervisor and safety officer immediately. Evacuate the area and begin cleaning up the spill as outlined in the safety manual. Dispose of contaminated materials properly.
- 7.11 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:
- 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.
- 7.12 Material Safety Data Sheets (MSDS) for all chemicals used are located on the laboratory intranet library and can be accessed through any connected laboratory computer.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

- 8.1 Water/wastewater 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 and/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 Gas 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 ⁶³Ni 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 144 Method:

- 9.1.1.1 Injector Temperature: 240° C
- 9.1.1.2 Detector Temperature: 320° C
- 9.1.1.3 Oven Program: Initial temperature at 160° C (no hold), ramp to 300° C at 8° C/min (hold 5.0 min)
- 9.1.1.4 Carrier Flow: approximately 30 cm/sec at the injection port
- 9.1.1.5 Column A: DB-35 (J&W 123-1933)
- 9.1.1.6 Column B: DB-XLB (J&W 123-1236)

9.1.2 Instrument 158 Method:

- 9.1.2.1 Injector Temperature: 210° C
- 9.1.2.2 Detector Temperature: 320° C
- 9.1.2.3 Oven Program: Initial temperature at 120° C (hold 0.0 min), ramp to 160° C at 20° C/min (hold 0.0 min), ramp to 300° C at 7° C/min (hold 5.0 min)
- 9.1.2.4 Carrier Flow: approximately 30 cm/sec at the injection point
- 9.1.2.5 Column A: RTX-CLP I (Restek 11140)
- 9.1.2.6 Column B: RTX-CLP II (Restek 11340)

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.

Note: Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

Note: Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

9.3 Microsyringes, glass, various sizes, gas-tight

9.4 Autosampler vials with PTFE-faced silicone septa, 2.0 mL

9.5 Amber vials, with PTFE-lined screw caps, 40 mL

- 9.6 Volumetric flasks, 10, 100, and 1000 mL class A
- 9.7 Analytical balance, capable of accurately weighing to 0.0001 g
- 9.8 Pasteur pipets, 2 mL, disposable, glass
- 9.9 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 inches 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.

11.0 CHEMICALS AND REAGENTS

- 11.1 Pesticide standards are purchased as certified solutions in heat-sealed ampoules.
- 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 1221/1154 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 Stock standards must be replaced after one year or sooner if comparison with calibration standards indicates a problem.

Note: Unopened purchased stock ampoules may be stored to the expiration date given by the manufacturer.

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 Aroclors 1016/1260 include 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 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 concentrations corresponding to the expected sample concentration range, that bracket the minimum detector response are 0.04, 0.1, 0.2, 0.4, 0.6, 0.8 and 1.0 ug/mL for 1016/1260. For TCMX/DCB, the concentrations are 0.0064, 0.0160, 0.0320, 0.0640, 0.0960, 0.1280, 0.160 ug/mL.

12.3.3 The lowest calibration standard is the minimum reporting limit.

12.3.4 The same 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 A standard of each Aroclor is required for pattern recognition. Assuming linearity has been demonstrated by the 1016/1260 calibration, a single-point calibration may be used for pattern recognition in samples. Prepare each single-point Aroclor standard at 0.40 ug/mL, including 0.064 ug/mL of 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 Element™
 - 12.4.6 Expiration dates must be no later than six months from the date of preparation for intermediate and working standards. 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 The solvent used
- 12.5 All standards preparation must be recorded in Element™. Refer to Attachment 23.2 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 diluted in accordance with TriMatrix SOP GR-09-105.
- 13.4 Extracts are cleaned up for sulfur in accordance with TriMatrix SOP GR-09-109.
- 13.5 Extracts are cleaned up with sulfuric acid in accordance TriMatrix SOP GR-09-110.
- 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 at or below the minimum reporting limit (500 ug/kg for waste dilution, 0.1 ug/L for water, 0.33 mg/kg for soil and 1.0 ug/wipe for wipes). A single-point 0.40 ug/mL calibration standard is used for pattern recognition of other Aroclors.
 - 14.1.1.1 Inject 1.0 µL of each calibration standard.
 - 14.1.1.2 Choose five peaks for quantitation, which are characteristic of the pattern.
 - 14.1.1.3 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 as follows:

$$\text{Calibration Factor (CF)} = \frac{\text{sum of the chosen peak responses}}{\text{Aroclor concentration, } \mu\text{g/mL}}$$

$$\text{Average Calibration Factor (CF}_{avg}\text{)} = \frac{\text{sum of calibration factors (CF}_{sum}\text{)}}{\text{number of calibration points}}$$

14.1.3 Calculate relative standard deviation (%RSD) of the CF_{avg} as follows:

$$\text{percent relative standard deviation (\%RSD)} = \frac{\text{standard deviation of } CF_{avg}}{CF_{avg}} \times 100$$

Note: The %RSD 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 (as indicated by reprocessing the lowest calibration point as a sample), use linear regression even if %RSD is acceptable.

14.1.4 If %RSD exceeds the method specified limit ($\geq 10\%$ for 608/ $> 20\%$ for 8082), perform instrument maintenance and run the calibration again. 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 .

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. Do not force 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 of the curve
 b = The intercept

14.1.5.2 Do not select linear 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 on both columns. For an Aroclor 1260 calibration curve example, refer to Attachment 23.3.

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)

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, determine the cause of failure, correct the problem 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 0.40 ug/mL of Aroclors 1016/1260 and surrogates (0.064 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:

$$\text{Percent Difference} = \frac{CF_{ccv} - CF_{avg}}{CF_{avg}} \times 100$$

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} and 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.

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 0.40 ug/mL Aroclor 1016/1260 CCV. Analyze the other Aroclor CCVs at 0.40 ug/mL at this time for pattern recognition.

15.2 After every 10 injections, run the 1016/1260 CCV to verify calibration remains in control.

15.2.1 Percent difference/drift must be $\pm 15\%$ to be acceptable for both Aroclors on both columns.

15.2.2 If % difference/drift is less than -15%, all samples run before and after the failing CCV must be re-injected on a system that is in control to be bracketed with acceptable QC.

15.2.3 If % difference/drift is more than +15%, samples run before the failing CCV with reportable results must be re-injected on a system that is in control.

15.2.4 If % difference/drift is more than +15% from the expected value, samples run before the failing CCV with results below the reporting limit do not need re-injected but only if the next CCV in the run sequence is greater than -15%.

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. To make a 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 Aroclor, forming a distinct pattern. To identify and report an Aroclor, the sample pattern of peaks must be qualitatively confirmed. Often, a pattern may be difficult to confirm 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 the sample matrix. Take extra care in identifying Aroclor 1221. Qualitatively determine samples as follows:

15.3.1.1 Use individual Aroclor injection associated with the sample run for the qualitative determination.

15.3.1.2 Print the sample run and Aroclor standard runs.

15.3.1.3 Perform an initial visual inspection of the sample run for obvious Aroclor patterns.

15.3.1.4 If an Aroclor pattern is suspected and/or observed on the hardcopy, pull the sample run up on the instrument computer and do a side-by-side comparison the suspected Aroclor, using the associated standard injection.

15.3.1.5 If the side-by-side comparison appears to be a qualitative match to the sample, perform an electronic overlay of the sample with the associated Aroclor standard.

15.3.1.6 If the electronic overlay confirms the suspected Aroclor, report quantitative results as the Aroclor. If the overlay does not match the suspected Aroclor, try the next closest Aroclor and continue until a match is determined.

15.3.1.7 If an exact match cannot be determined and/or if multiple Aroclors are observed, causing problems with pattern recognition, report the closest matching Aroclor with narration fully detailing the problems encountered.

15.3.1.8 All peaks used in the qualitative determination must elute within the retention time windows of both columns.

15.3.1.9 All qualitative results must be confirmed by peer review prior to quantitative analysis.

15.3.2 Aroclors can be grouped into two groups. Early eluting Aroclors 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 23.8 (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 23.8) except for 1221. 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.

15.3.3 Late eluting Aroclors:

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 23.8).

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 23.8) if early Aroclors are present. Do not use disproportionate peaks, to avoid matrix interference.

15.3.3.3 Quantitation of other than the 1016/1260 Aroclors must be against the appropriate Aroclor calibration.

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 23.8). 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 on both columns.
- 15.3.4.3 Quantitation of other than the 1016/1260 Aroclors must be against the appropriate Aroclor 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 established retention time window for that peak.
- 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 CCV must elute within the retention time window on both columns. Samples run in association with any subsequent CCV eluting outside the retention time window for either column must be re-analyzed.
- 15.4.5 Update retention time windows whenever a new column is installed.
- 15.5 Example chromatograms showing 1016/1260 Aroclor patterns on each column are provided in Attachments 23.9 – 23.12.
- 15.6 All qualitative pattern matches must be quantitatively confirmed by retention times and by looking at the second column result.
- 15.6.1 The secondary quantitation must meet all QC criteria as required for the primary column, including calibration and retention times. Evaluate the agreement between results by calculating the relative percent difference.
- 15.6.2 Calculate relative percent difference (RPD) 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.

$$RPD = \frac{|R_1 - R_2|}{R_1 + R_2} \times 200$$

- 15.6.3 If one result is significantly higher (if RPD is greater than 40%), check to see if obviously overlapping peaks are 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 the extract 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.
- 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:

$$\text{Wipe Concentration (in total ug)} = (\text{ug / mL})(V_e)(DF)$$

Soil Samples:

$$\text{Sample Concentration (in mg/kg)} = \frac{(\text{ug/mL})(V_e)(DF)}{(V_s)(PS)} \times 1000$$

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 23.6 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 Blanks, surrogates and matrix spikes must be included as part of quality control.

Note: If insufficient sample is received to perform a matrix spike/matrix spike duplicate, the report must be narrated as follows:

GN006 Due to insufficient sample volume received, no matrix QC is available with this sample batch.

- 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 dechlorobiphenyl 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 exact 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 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:

$$\text{Upper Control Limit (UCL)} = p + 3s$$
$$\text{Lower Control Limit (LCL)} = p - 3s$$

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.

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) in accordance with Section 15.6.2.
- 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 LRB 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.
- 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 analysis 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.
- 19.2 Prepare four blank spikes of 1016/1260 using a spiking solution from a source other than that used to calibrate the analytical instrument. Prepare for analysis by going through all extraction steps in the procedure.
 - 19.2.1 The IDC must include running a minimum 5-point curve for 1016/1260 and running a single-point standard for all other Aroclors.
 - 19.2.2 Then, have the trainer prepare a single-blind random dilution of each of the nine other Aroclors throughout the calibration range. These nine unknowns are to be qualitatively evaluated by the trainee to report which Aroclor each is. The 1016/1260 blank spikes are to be quantitatively evaluated.
 - 19.2.3 All nine unknown Aroclors must be identified correctly and must recover within 80 - 120%.
 - 19.2.4 If any is misidentified, the IDC fails. Additional training and practice must be undertaken in Aroclor identification and nine fresh dilutions at random concentrations made and analyzed.

- 19.2.5 All nine Aroclors must be qualitatively identified and recovered correctly for training to be considered complete.
- 19.3 For the quantitative part of the IDC study, after preparing, analyze the four 1016/1260 extracts.
- 19.4 Input results to the IDC spreadsheet located on the laboratory intranet library to calculate percent recovery and relative standard deviation for each spiked analyte.
- 19.4.1 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 prepare waste samples by this procedure.
- 19.4.2 If any analyte fails any acceptance limit, locate and correct the problem then repeat the study successfully for the failed analyte.
- 19.5 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.
- 19.6 Samples may not be prepared by the analyst until a successful IDC study has been completed.
- 19.7 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 extracting an acceptable blind performance testing sample.
- 16.7.3 By exclusively preparing a method detection limit (MDL) study. The last four results of the study may be used.
- 19.8 A Method Detection Limit (MDL) study must be performed annually for both extraction procedures 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.

- 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 Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*, 3rd Edition, Final Update IV, February, 2007, Method 8082A, "Polychlorinated Biphenyls (PCBs) by Gas Chromatography", Revision 0, December, 1996
- 22.2 *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 808, Organochlorine Pesticides and PCBs*

23.0 ATTACHMENTS/APPENDICES

- 23.1 Default Reporting Limits
- 23.2 Standards Log Example
- 23.3 Sample of Calibration Curves for PCBs 1016/1260
- 23.4 Preparation Batch Report Example
- 23.5 Analysis Sequence Report Example
- 23.6 Sample Page of Instrument Run Logbook
- 23.7 Method Detection Limit Study Example
- 23.8 Key Peaks for Arochlor Identification
- 23.9 1016/1260 on RTX CLP Column (Instrument 158)
- 23.10 1016/1260 on RTX CLP2 Column (Instrument 158)
- 23.11 1016/1260 on DB-35 Column (Instrument 144)
- 23.12 1016/1260 on DB-XLB Column (Instrument 144)

**Attachment 23.1
 Default Reporting Limits**

**Attachment 21.1
 Default Reporting Limits**

<u>Compound</u>	<u>Water (ug/L)</u>	<u>Soil (mg/kg)</u>
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

<u>Compound</u>	<u>Wipes (ug)</u>	<u>Waste (mg/kg)</u>
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

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SOP Name: Polychlorinated Biphenyls (PCB) by Gas Chromatography
 EPA Method 608, SW-846 Method 8082A
 SOP Number: GR-03-128 Page 24 of 34

Revision Number: 2.7
 Date Revised: 2/17/12
 Date Initiated: 4/2/98

**Attachment 23.2
 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 W

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

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Reviewed By _____

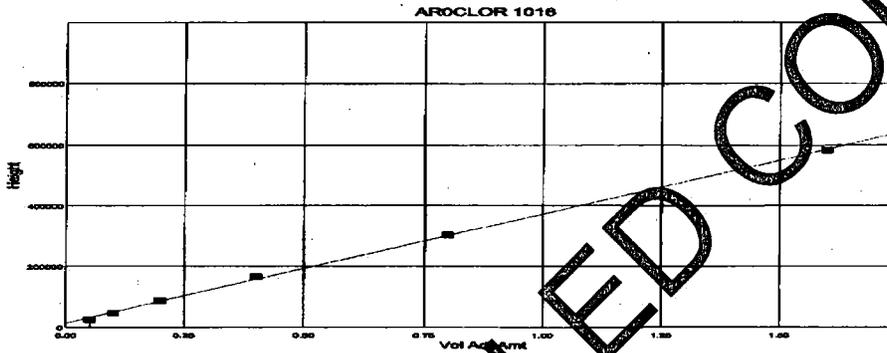
Date _____

Attachment 23.3
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 = (14080.795690) + (388083.114083)X

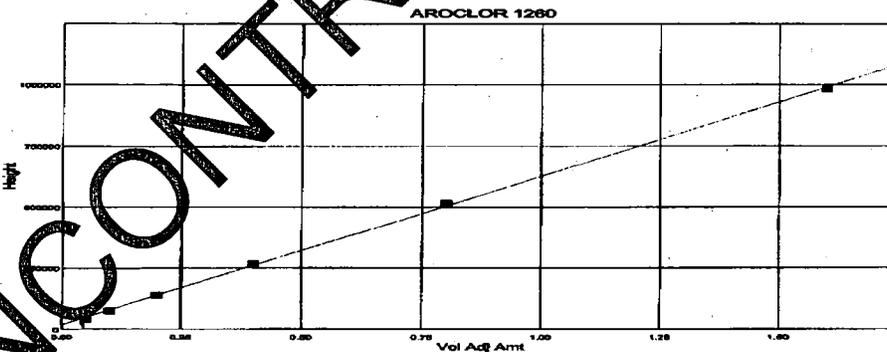


Level Name	Observed X-Value	Calculated Y-Value	Delta	SDIFF.	Observed Y-Value	Calculated Y-Value	Delta	SDIFF.
0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
0.10000	0.052497	0.052497	0.00000	0.00000	24991.254	24991.254	0.00000	0.00000
0.20000	0.104994	0.104994	0.00000	0.00000	49982.508	49982.508	0.00000	0.00000
0.40000	0.209988	0.209988	0.00000	0.00000	99965.016	99965.016	0.00000	0.00000
0.80000	0.419976	0.419976	0.00000	0.00000	199930.032	199930.032	0.00000	0.00000
1.60000	0.839952	0.839952	0.00000	0.00000	399860.064	399860.064	0.00000	0.00000

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) $R^2 = 0.999451$
 Calibration Curve = (18222.516877) + (407243.479103)X



Level Name	Observed X-Value	Calculated Y-Value	Delta	SDIFF.	Observed Y-Value	Calculated Y-Value	Delta	SDIFF.
0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
0.10000	0.036871	0.036871	0.00000	0.00000	40437.606	40437.606	0.00000	0.00000
0.20000	0.073742	0.073742	0.00000	0.00000	80875.212	80875.212	0.00000	0.00000
0.40000	0.147484	0.147484	0.00000	0.00000	161750.424	161750.424	0.00000	0.00000
0.80000	0.294968	0.294968	0.00000	0.00000	323500.848	323500.848	0.00000	0.00000
1.60000	0.589936	0.589936	0.00000	0.00000	647001.696	647001.696	0.00000	0.00000

SOP Name: Polychlorinated Biphenyls (PCB) by Gas Chromatography
 EPA Method 608, SW-846 Method 8082A
 SOP Number: GR-03-128

Revision Number: 2.7
 Date Revised: 2/17/12
 Date Initiated: 4/2/98

**Attachment 23.4
 Preparation Batch Report Example**

TriMatrix Laboratories, Inc.

PREPARATION BATCH # 0705430 Page 1 of 1

Printed: 9/14/2008 4:20:19PM

Semivolatiles GC: Water, 3510C Liquid-Liquid Extraction
 Surrogate #1 = 7040593 (Pre-Prep)

Batch Comments: (none)

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	mL Surrogate	Source ID	Spike ID	mL Spike	Client / QC Type	Batch Comments
0705430-BLK1		May-18-07 09:34	ASC	1000	2	1000				BLANK	
0705430-B51		May-18-07 09:34	ASC	1000	2	1000		6120832	1000	LCS	
0705288-01	D	May-18-07 09:34	ASC	1000	2	1000					
0705322-01	H	May-18-07 09:34	ASC	1080	2	1000					
0705322-02	I	May-18-07 09:34	ASC	1080	2	1000					
0705322-03	E	May-18-07 09:34	ASC	1080	2	1000					
0705322-04	G	May-18-07 09:34	ASC	1080	2	1000					
0705322-06	E	May-18-07 09:34	ASC	1080	2	1000					

Comments:	Analysis Name:
-----------	----------------

Tech: JnMstr@pt

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**Attachment 23.5
 Analysis Sequence Report Example**

TriMatrix Laboratories, Inc. ANALYSIS SEQUENCE **7052932** Page 1 of 3 Printed: 9/14/2007 4:24:07 PM

Semivolatiles GC, Water, May-29-07
 Instrument = 144, Calibration = 7F:04002

<i>Sequence Analysis:</i> 608 PCBs (std 7 aroclors) 8082 PCBs (master list)	608 PCBs (Tonawanda) 8082 PCBs (std 7 aroclors)
---	--

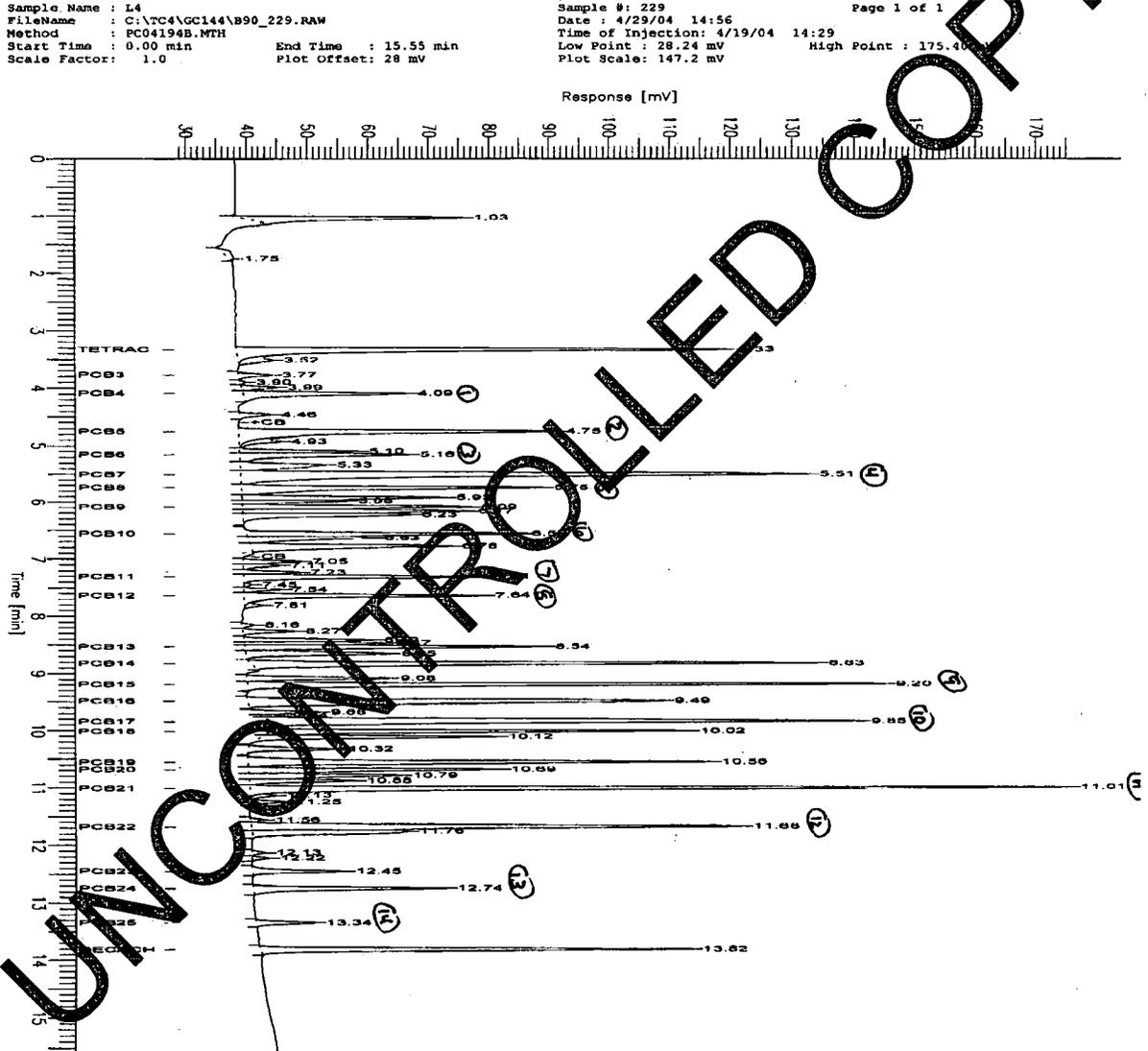
Lab Number	Analysis	Contains	STD ID	ISTD ID	Client QC Type	Extraction Comments
7052932-ICV1	QC		7030266		INITIAL CAL CHECK	
7052932-ICV2	QC		7030949		INITIAL CAL CHECK	
7052932-ICV3	QC		7040212		INITIAL CAL CHECK	
7052932-ICV4	QC		7030269		INITIAL CAL CHECK	
7052932-ICV5	QC		7030271		INITIAL CAL CHECK	
0705288-04	8082 PCBs (master list)	A 02				
0705392-01	8082 PCBs (std 7 aroclors)	C 02				
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				
0705322-01	8082 PCBs (std 7 aroclors)	H 01				
0705322-02	8082 PCBs (std 7 aroclors)	I 01				
0705322-03	8082 PCBs (std 7 aroclors)	J 01				
7052932-CCV1	QC		7030271		CALIBRATION CHECK	
0705322-04	8082 PCBs (std 7 aroclors)	K 01				
0705322-05	8082 PCBs (std 7 aroclors)	E 01				
7052932-CCV2	QC		7030271		CALIBRATION CHECK	
0705651-BLK1	QC				BLANK	
0705651-BS1	QC				LCS	

Comments:	Analyst Initials:
-----------	-------------------

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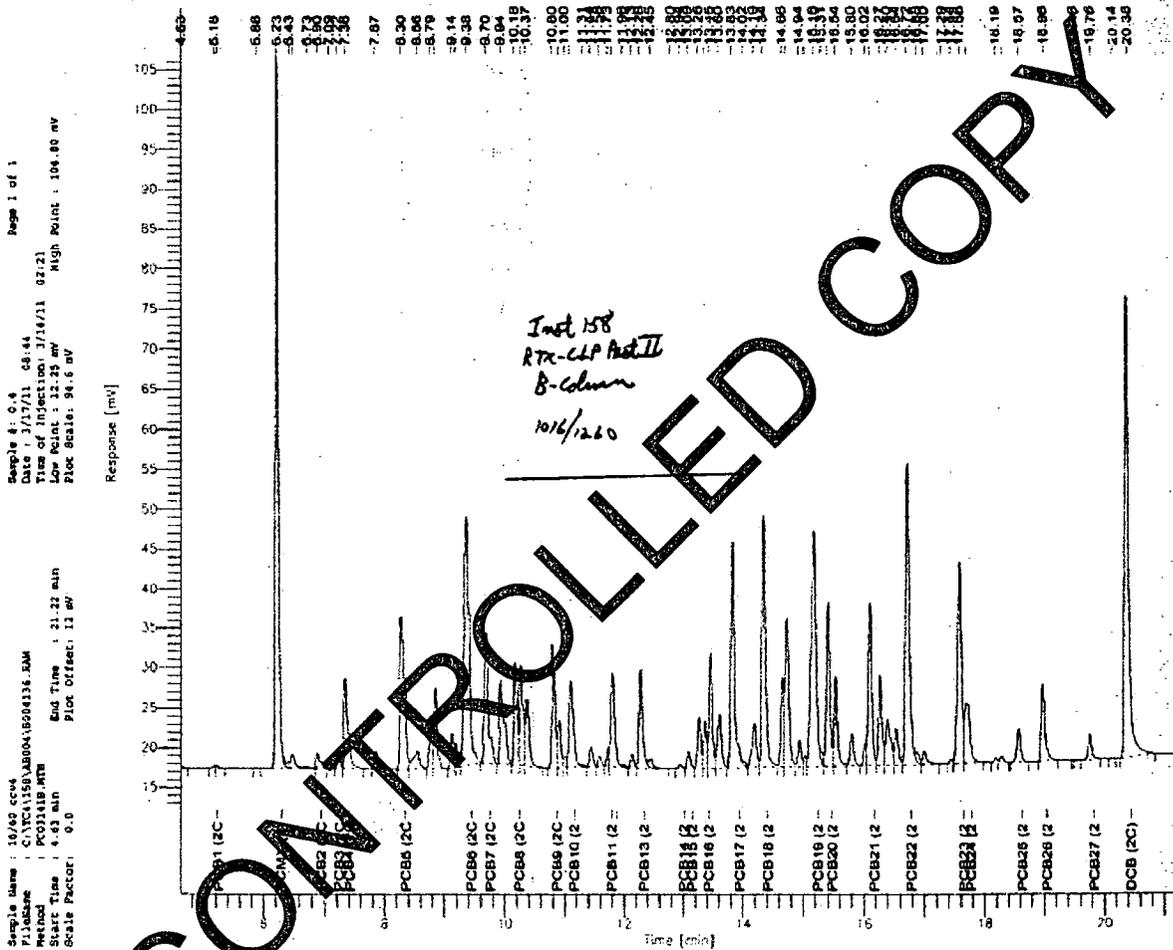
Attachment 23.8
Key Peaks for Aroclor Identification

Chromatogram



Attachment 23.10
1016/1260 on RTX CLP2 Column (Instrument 158)

Chromatogram



Sample Name : 10/AP ccv4
File Name : C:\YCA\158\AB004\B004136.DAM
Method : PC03118.MTH
Start Time : 4.43 min
Scale Factor: 0.0
End Time : 21.22 min
Plot Offset: 13 mV

Sample #: 0.4
Date : 3/17/11 08:44
Time of Injection: 3/14/11 02:21
Low Point : 12.25 mV
High Point : 104.80 mV
Plot Scale: 94.6 mV

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SOP Name: Polychlorinated Biphenyls (PCB) by Gas Chromatography
EPA Method 608, SW-846 Method 8082A
SOP Number: **GR-03-128**

Revision Number: 2.7
Date Revised: 2/17/12
Date Initiated: 4/2/98

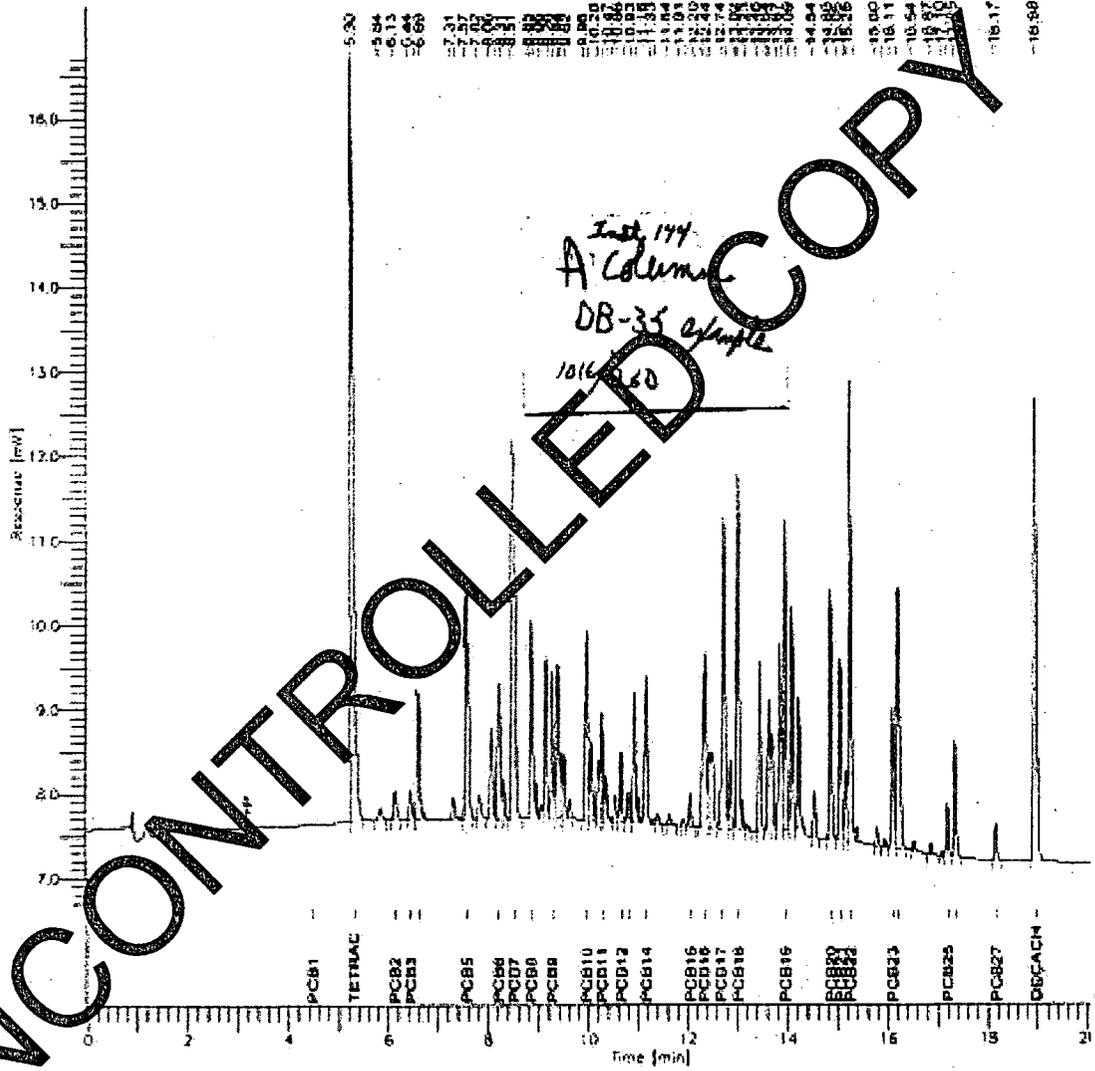
Attachment 23.11
1016/1260 on DB-35 Column (Instrument 144)

Chromatogram

Page 1 of 1
Sample #: 1016/1260
Date: 3/13/11 08:22
Time of Injection: 3/13/11 22:41
Loop Point: 6.64 mL
Split Point: 16.73 mL
Split Ratio: 10:1

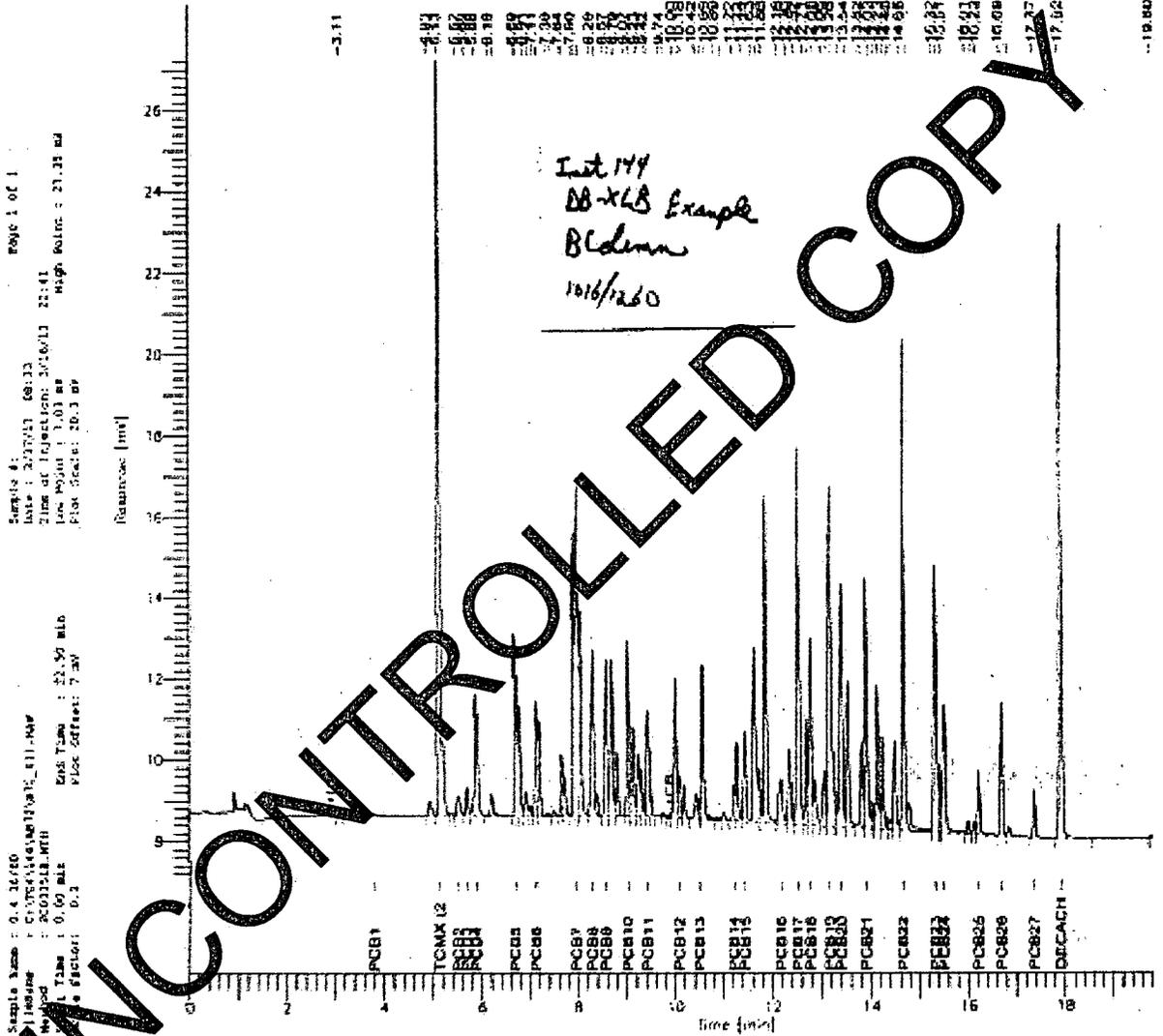
Sample Name: 2-4-10/10
Injection: 0.1 µL
Carrier Gas: N₂
Flow Rate: 1.00 mL/min
Split Ratio: 10:1
Split Point: 16.73 mL

Sample Name: 2-4-10/10
Injection: 0.1 µL
Carrier Gas: N₂
Flow Rate: 1.00 mL/min
Split Ratio: 10:1
Split Point: 16.73 mL



Attachment 23.12
1016/1260 on DB-XLB Column (Instrument 144)

Chromatogram





STANDARD OPERATING PROCEDURE

Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry

EPA Method 625 SW-846 Method 8270C

APPROVALS:

Area Supervisor: *John Blouin*
John L. Blouin

Date: 6-23-12

QA Officer: *Tony Boocher*
Tony Boocher

Date: 6-29-12

Laboratory President: *Douglas E. Kriscunas*
Douglas E. Kriscunas

Date: 6-29-2012

Procedure Number: GR-04-103
Revision Number: 5.8

Date Initiated: 12/1/98
Effective Date: 7/2/12

Date Revised: 6/22/12
Pages Revised: All

By: Rick D. Wilburn

Total Number of Pages: 49

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
_____	_____	_____
_____	_____	_____
_____	_____	_____

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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. Benzilene 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*

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 $\leq 20\%$ 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 Review*, latest revision
- 5.8 TriMatrix Laboratories 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. 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 grade 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 Wear a laboratory coat and approved safety glasses while in the laboratory except when sitting at a computer terminal entering data.
- 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.
- 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 exposure 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 Waste samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-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 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 0 - 6° 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 section 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 50 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: 1.0 uL
- Carrier Gas: ultra high purity helium at 6 psi (~ 1 to 2 mL/minute)

9.1.2 The Agilent 5973 and 5975 Mass Selective Detector (MSD) are 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 50 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 6890/7890 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 Injectors

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 0.32 mm 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.

NOTE: Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

NOTE: Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

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-d₄, benaphthene-d₁₀, phenanthrene-d₁₀, chrysene-d₁₂, naphthalene-d₈, and perylene-d₁₂. 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 50 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-6, 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 0 - 6° C.

Surrogate	Mass (g)	Dilution (in methanol)	Stock (mg/L)	Dilution #2 (mL:mL)	Final Concentration
Decafluorobiphenyl	0.5	250 mL	2000	100:2000	100 ug/mL
o-terphenyl	0.5	250 mL	2000	100:2000	100 ug/mL
Nitrobenzene-d ₅	0.5	250 mL	2000	100:2000	100 ug/mL
2-Fluorobiphenyl	0.5	250 mL	2000	100:2000	100 ug/mL
2,4,6-Tribromophenol	1.0	250 mL	4000	100:2000	200 ug/mL
Phenol-6	1.0	250 mL	4000	100:2000	200 ug/mL
2-Fluorophenol	1.0	250 mL	4000	100:2000	200 ug/mL

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 auto sampler.
- 14.3 Each GC/MS system is hardware tuned to meet Table 3 or Table 3A criteria for a 50 ng, 1.0 uL DFTPP injection. No analysis may begin until these criteria are met.
- 14.3.1 The following evaluation sequence must be used to determine DFTPP tune performance.
- 14.3.1.1 All DFTPP tunes must be evaluated using the instrument software autofind DFTPP 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. The ion trap DFTPP is also evaluated this way, but there is no autofind feature, it is done manually.
- 14.3.1.2 If the above does not give an acceptable DFTPP tune, then corrective action is required. The first step is to reinject and reanalyze the same DFTPP standard. Next, prepare a new DFTPP 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.
- 14.3.2 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%. Most of the instruments have software that calculates degradation (Attachment 23.16). When available, include this form in all data packages. If the software does not perform this check, print off a chromatogram displaying the DDD, DDE, and DDT peaks, record the peak areas, and input them into the following equation:

$$\% \text{ Degradation} = \frac{\text{Area}_{\text{DDD}} + \text{Area}_{\text{DDE}}}{\text{Area}_{\text{DDT}} + \text{Area}_{\text{DDE}} + \text{Area}_{\text{DDD}}} \times 100$$

- 14.3.3 Transfer the calculated result (not simply <20) to the daily run logbook.
- 14.3.4 Pentachlorophenol and benzidine must be present at their normal responses, with minimal peak tailing. Calculate a tailing factor using the pentachlorophenol and benzidine total ion chromatogram. Include tailing factor results with the DFTPP instrument printout. 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 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 (0.1 to 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 0.05 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:

$$C_s = [(A_s \cdot C_{is}) / A_{is}] - b / a$$

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:

$$[(A_s \cdot C_{is})/A_{is}] = a \cdot C_s^2 + b \cdot C_s + c$$

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:

$$[(A_s \cdot C_{is})/A_{is}] = a \cdot C_s^3 + b \cdot C_s^2 + c \cdot C_s + d$$

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" 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:

$$\% \text{ Difference} = \frac{RF_v - RF_m}{RF_m} \times 100$$

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:

$$\% \text{ Drift} = \frac{C_c - C_1}{C_1} \times 100$$

where:

C₁ = theoretical CCC standard concentration
C_c = measured concentration using appropriate regression fit.

14.6.4 If percent difference or drift for every CCC is less than $\pm 20\%$, the initial calibration remains valid. If any CCC compound differs from the initial calibration curve by greater than $\pm 20\%$, 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 $\leq 25\%$, with the exception of poor performing analytes, which can have up to four outside of $\leq 25\%$, but must be $\leq 40\%$. 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 $\leq 20\%$.

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 midpoint standard level of the most recent initial calibration sequence, 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 18.9.
- 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 $\pm 30\%$. For example, an ion with 50% abundance in standard spectra, means the corresponding sample abundance must be between 20% and 80%.
- 16.1.1.3 All spectra, with the following two exceptions, will be printed and stored with the raw data. Exception #1: Peer review will not be required, nor will a hard copy of the spectra be retained, if the false positive is based on a poor retention time match (making spectral match irrelevant). Exception #2: Peer review will not be required, nor will a hard copy of the spectra be

retained, for analytes detected below the reporting limit (when reporting of estimated results is not required). All remaining spectra must be initialized by the peer performing the review.

- 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-XX-XXXX
Unknown Ketone	X-XXX-XX
Unknown Acid	XX-XX-XXXX
Unknown Hydrocarbon	XXX-XXX
Unknown Glycol Ethers	XX-XX-X
Unknown Substituted Benzenes	XXXXX
Hydrocarbons (Substituted 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 $\pm 20\%$. 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 70 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:

$$\text{Concentration in ug/L} = \frac{(A_x)(I_s)(V_t)(Dil)}{(A_{is})(RF)(V_o)}$$

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:

$$\text{Concentration in mg/kg} = \frac{(A_x)(I_s)(V_t)(Dil)}{(A_{is})(RF)(W_s)(D)}$$

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. In addition, the appropriate LIMS qualifier must be attached to all manual integrations.

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-Dichlorobenzene), jagged analyte peak shapes split by the integrator, and analytes like benzidine that have significant peak-tailing.

NOTE: Under no circumstances must manual integration be applied solely for the purpose of meeting quality control. Peak shaving and peak enhancing are strictly prohibited.

16.3.4 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 Data System. 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 The following analyte specific LIMS qualifiers must be attached as appropriate:

SV001

3-Methylphenol cannot be resolved from 4-Methylphenol due to chromatographic limitations. The reported result could be 3-Methylphenol, 4-Methylphenol, or a combination of both isomers.

SV002

Benzo(b)fluoranthene could not be resolved from Benzo(k)fluoranthene due to sample matrix interference. The reported result could be Benzo(b)fluoranthene, Benzo(k)fluoranthene, or a combination of both isomers.

SV003

The result reported as Diphenylamine could be Diphenylamine, N-Nitrosodiphenylamine, or a combination of both isomers. N-Nitrosodiphenylamine decomposes during analysis and cannot be distinguished from Diphenylamine.

SV008

Hexachlorophene is an unstable compound and was not included in the calibration standards. Because hexachlorophene (CAS 70-30-4) is a required Appendix IX analyte, a mass search was performed. The presence of this analyte was not detected in this sample.

17.2.3 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 initialed. 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, spectra 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 VIIs.
- 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 current 12-hour continuing calibration standard.

NOTE For DoD projects the internal standard retention times and areas are compared to the midpoint standard of the most recent 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 re-analysis 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:

$$\text{Upper Control Limit (UCL)} = p + 3s$$

$$\text{Lower Control Limit (LCL)} = p - 3s$$

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 Batches must be opened and closed every 24 hours. Each batch of up to 20 samples must include an extraction blank (BLK), blank spike (BS), matrix spike (MS) and matrix spike duplicate (MSD).

Note: If insufficient sample is received to perform a matrix spike/matrix spike duplicate, the following LIMS narrative must be attached to the batch:

GN006 – Due to insufficient sample volume received, no matrix QC is available with this sample batch.

18.5.2 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. Batches are matrix specific, must be opened and closed daily, and can contain no more than 20 samples.

NOTE: For State of Wisconsin samples and where project specifications require, all target analytes must be added for associated spikes, spike duplicates, and BSs.

18.5.3 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%.

18.5.4 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 $R \pm 3SD$. 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.

18.5.5 Calculate percent recovery as follows:

$$\% \text{ recovery} = \frac{(A_{\text{spk}} - A_{\text{smpl}})}{\text{SPK}} \times 100$$

where:

A_{spk} = The spiked sample concentration, in ug/L or mg/kg

A_{smpl} = The non-spiked sample concentration, in ug/L or mg/kg

$$\text{SPK} = \left(\frac{\text{concentration of spiked standard in ug/L} \times \text{mL spiked}}{\text{initial sample volume in L}} \right)$$

18.5.6 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

18.5.7 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.

18.5.8 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 A Method Detection Limit (MDL) study must be extracted annually in accordance with GR-10-125 for each sample matrix/extraction technique.

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 initial 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 on 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 *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

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- 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 Additional 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
- 23.9 Table 7 Demonstration of Capability Study Example
- 23.10 Figure 1 Pentachlorophenol Tailing Factor
- 23.11 Figure 2 Continuing Calibration Standard (10 ug/mL) Total Ion Chromatogram, Varian Ion Trap
- 23.12 Figure 2A Continuing Calibration Standard (10 ug/mL) Total Ion Chromatogram, Agilent 5973 MSD
- 23.13 Figure 3 Standards Form Example
- 23.14 Figure 4 Analytical Run Logbook Example
- 23.15 Method 8270C Selected Ion Monitoring (SIM) instructions
- 23.16 4,4'-DDT Degradation Result Example

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, 255	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	111	64	1
Nitrobenzene-d ₅ (surr.)	11.38	127+128 (128)*	82, 54	2
Phenol-d ₆ (surr.)	9.13	91		1
2,4,6-Tribromophenol (surr.)	19.33	330	332, 141	3
Terphenyl-o (surr.)	21.03	239	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	23.06	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-Bromobenzyl phenyl ether	19.73	248	250, 141	4
Butyl benzylphthalate	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

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, 179	6
Dibenzofuran	18.20	168	139	3
Di-n-butylphthalate	21.23	149	150, 164	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	62	164, 98	2
Diethylphthalate	18.59	149+173 (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	17.26	165	63, 89	3
1,2-Diphenylhydrazine	19.71	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-Methylnaphthalene	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

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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, 15	2
4-Nitrophenol	18.02	65	109, 65	3
N-Nitrosodimethylamine	4.58	73+74 (74)**	42	1
N-Nitrosodiphenylamine	19.08	168	168, 167	4
N-Nitrosodipropylamine	10.95	70	42, 101, 1	1
Pentachlorophenol	20.18	266	264, 268	4
Phenanthrene	20.54	179	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	15.76	196	198, 200	3

I.S. = Internal Standard
 surr. = Surrogate
 * retention time provided for example only.
 ** for MSD

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**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 Std
Acenaphthene-d ₁₀ (I.S.)	14.801	164	164,162	3
Chrysene-d ₁₂ (I.S.)	21.72	240	238,241	5
Dichlorobenzene-1,4-d ₄ (I.S.)	7.935	152	150,75	1
Naphthalene-d ₈ (I.S.)	10.674	136	108	2
Perylene-d ₁₂ (I.S.)	23.503	264	263,132	6
Phenanthrene-d ₁₀ (I.S.)	18.176	188	184,160	4
1,1'-Biphenyl	13.478	154	153,152	3
1,2,4,5-Tetrachlorobenzene	12.738	216	214,181	3
1,3-Dinitrobenzene	14.3	168	-	2
1,4-Naphthoquinone	17.842	158	102,76	2
1-Methylnaphthalene	2.569	142	141,115	3
1-Naphthylamine	15.41	143	115	3
2,3,4,6-Tetrachlorophenol	15.611	232	230,131	3
2,3,5,6-Tetrachlorophenol	15.506	232	230,131	3
2,6-Dichlorophenol	10.884	162	164,63	2
2-Acetylaminofluorene	21.326	181	223,152	4
2-Chloroaniline	9.759	127	92,65	2
2-Ethoxyethanol	3.132	59	-	1
2-Naphthylamine	15.608	143	115	3
2-Picoline	4.551	93	66	1
2-sec-Butyl-4,6-dinitrophenol	18.172	211	163,147	4
3,3-Dimethylbenzidine	21.046	212	213,96	4
5-Methylcholanthrene	23.844	268	252	6
4-Aminobiphenyl	17.805	169	168	3
6-Nitroquinoline-1-oxide	19.426	190	160	4
5-Nitro-o-toluidine	16.109	152	77	3
7,12-Dimethylbenz(a)anthracene	22.888	256	239,241	5
a,a-Dimethylphenethylamine	10.234	58	91	2
Acetophenone	8.792	105	77,120	1
Aniline	7.299	93	66	1
Aramite™	20.574	185	135,175	4
Atrazine	17.762	200	215	4
Benzaldehyde	7.095	105	106,77	1

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**Attachment 23.2
 Table 1A (continued)
 Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and
 Secondary Ions**

Benzidine	20.239	184	92	5
Bis(2-chloroethoxy)ethane	11.91	63	93,107	2
Caprolactam	11.671	55	113,85	2
Carbazole	18.619	167	161,139	4
Chlorobenzilate	20.714	251	139,111	4
Diallate	17.117	86	234	3
Dicyclohexylphthalate	21.766	149	167,55	5
Dimethoate	17.491	87	125,93	3
Disulfoton	18.207	88	89,93	4
Ethyl methacrylate	4.085	69	100	1
Ethyl methanesulfonate	6.422	79	109	1
Famphur	20.999	218	125,93	4
Hexachlorophene	27.48	196	198,209	6
Hexachloropropene	10.31	213	215,211	2
Isodrin	19.769	193	66,263	4
Isosafrole	13.329	162	104	2
Kepone	21.075	272	274,237	4
Methapyrilene	19.571	58	97	4
Methyl methacrylate	3.047	69	41,100	1
Methyl methanesulfonate	5.233	80	65	1
N-Nitrosodiethylamine	5.88	102	42,44	1
N-Nitrosodipropylamine	11.638	84	57,41	2
N-Nitrosomethylmethylethylamine	4.737	88	42,43	1
N-Nitrosopiperidine	8.771	56	-	1
N-Nitrosopyrrolidine	9.394	114	55	1
N-Nitrosopyrrolidine	8.677	100	41	1
o,o-dithiophosphorothioate	10.071	121	198,97	1
o-Toluidine	8.777	106	79	1
p-(Dimethylamino)azobenzene	20.655	120	225,77	4
Parathion, Ethyl	19.478	109	97, 291	4
Parathion, Methyl	18.831	109	125,263	4
Pentachlorobenzene	15.101	250	252,108	3
Pentachloroethane	7.29	167	119	2
Pentachloronitrobenzene	17.77	237	214,142	3
Phenacetin	17.211	108	137,179	3
Phorate	17.141	75	121,97	2
p-Phenylenediamine	11.621	108	80	2
Pronamide	17.98	173	175,145	3

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**Attachment 23.2
 Table 1A (continued)**

**Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and
 Secondary Ions**

Pyridine	3.587	79	52	1
Safrole	12.105	162	104	2
Sulfotepp	16.884	322	97, 102	3
sym-Trinitrobenzene	17.077	75	113	3
Thionazin	16.08	97	107, 143	3

I.S. = Internal Standard

***retention time provided for example only**

****Aramite chromatographs into three peaks**

***** For MSD**

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**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	111-44-1	5	0.33
Bis(2-chloroisopropyl)ether	39628-37-9	5	0.33
Bis(2-ethylhexyl)phthalate	117-81-7	5	0.33
4-Bromophenyl phenyl ether	100-55-2	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

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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	71-47-4	5	0.33
Hexachloroethane	67-72-1	5	0.33
Indeno(1,2,3-cd)pyrene	93-39-5	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

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**Attachment 23.3
 Table 2 (continued)
 Default Reporting Limits**

Semivolatiles	CAS Number	Default Reporting Limits**	
		Surface/Ground/ Wastewater ug/L	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.

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**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	99-65-3	5	0.33
Ethyl methacrylate	97-63-2	50	2.0
Ethyl methanesulfonate	62-50-0	10	1.0
Hexachlorophene	70-30-4	***	***
Hexachloropropene	888-71-7	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 oxide	56-57-5	500	20
N-Nitrosodimethylamine	924-16-3	20	0.90
N-Nitrosodimethylamine	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

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**Attachment 23.4
 Table 2A (continued)
 Additional Default Reporting Limits**

Semivolatiles	CAS Number	Default Reporting Limits**	
		Surface/ Ground Water ug/L	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	95-94-8	10	0.50
2,3,4,6-Tetrachlorophenol	53-90-2	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 Default Criteria)**

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

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**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	

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Attachment 23.8
 Table 6
 Method Detection Limit Study Example



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	0.46	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	0.423	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	0.43	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	0.780	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	0.55	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	0.77	0.5	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	0.600	0.700	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	0.45	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	0.47	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	0.36	0.35	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	0.41	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	75%	0.194	0.611

Attachment 23.9
Table 7
Demonstration of Capability Study Example



SEMI-VOLATILE LABORATORY
DEMONSTRATION OF CAPABILITY FOR SCOTT J. PUGH

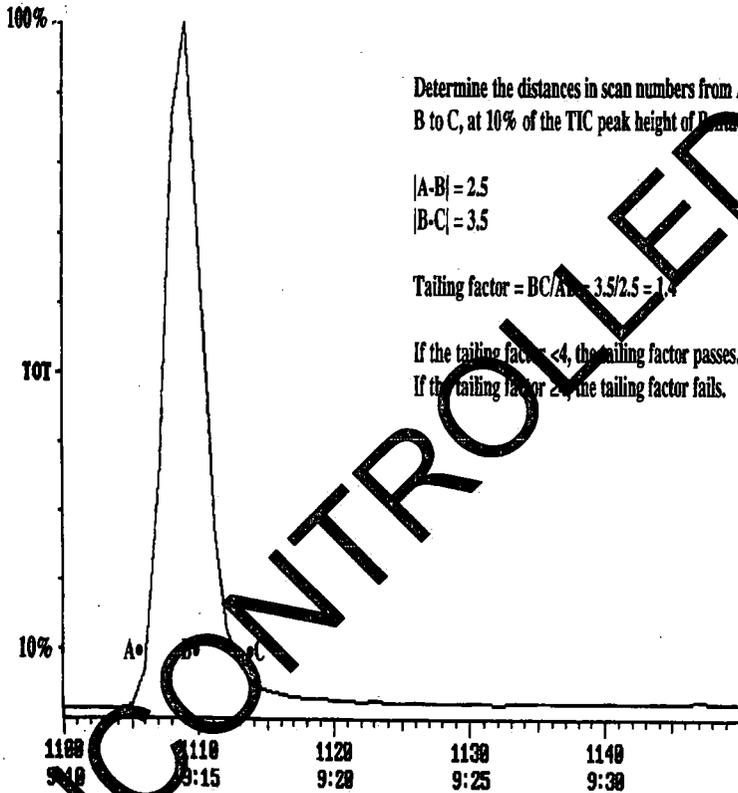
Parameter	Date Analyzed	Method	Inst. Number	Units	Amount Spiked	Cert. #1 Amount Found	Cert. #2 Amount Found	Cert. #3 Amount Found	Cert. #4 Amount Found	Average Percent Recovery	Percent Recovery Window	Pass/Fail Percent Recovery	Percent RSD	Percent RSD Window	Percent RSD Pass/Fail	Overall Pass/Fail
Phenol	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	8.4000	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	8.4000	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	7.0200	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	9.5800	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	8.0300	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	97.0%	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	71.8%	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%	35 -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	1.1%	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

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Attachment 23.10
Figure 1
Pentachlorophenol Tailing Factor

Chromatogram Plot C:\SATURN\DATA\DFI1210E Date: 12/10/98 21:08:21
 Comment: INSTRUMENT 138
 Scan: 1150 Seg: 1 Group: 0 Retention: 9:35 RIC: 138853 Masses: 40-356
 Plotted: 1100 to 1150 Range: 1 to 2016 100% = 17694537



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

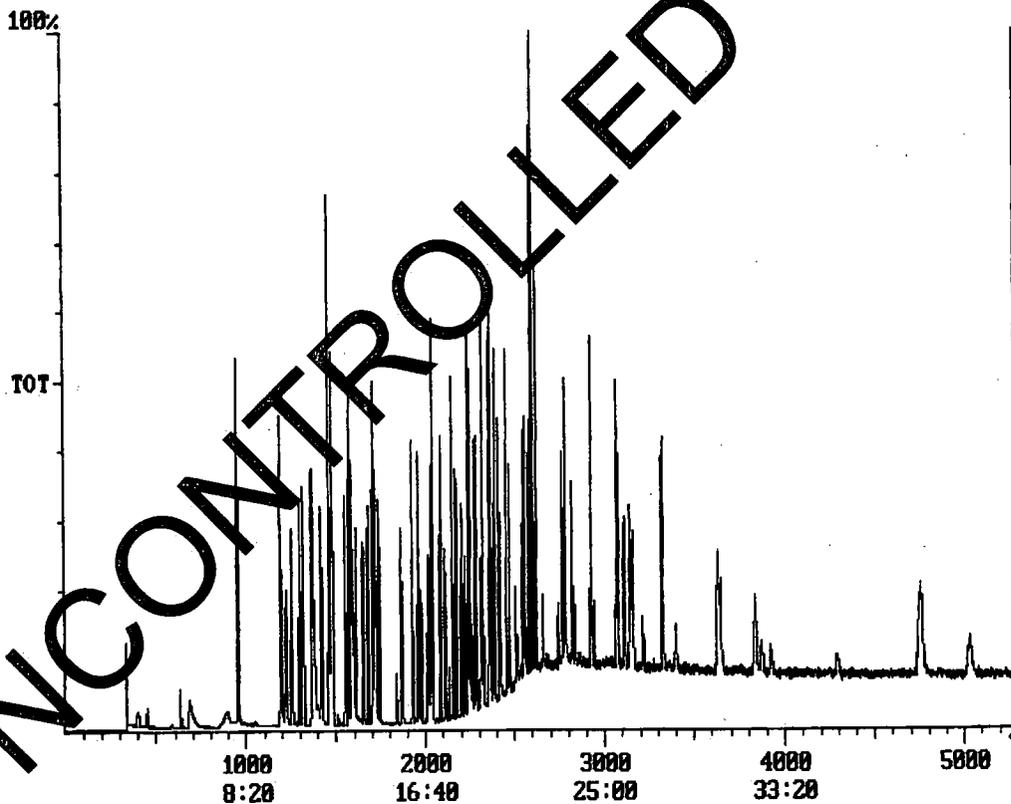
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Attachment 23.11
Figure 2
Continuing Calibration Standard (10 ug/mL)
Total Ion Chromatogram, Varian Ion Trap

Chromatogram Plot C:\SATURN\DATA\10C0428A Date: 04/28/04 12:04:14
Comment: INSTRUMENT 133
Scan: 5280 Seg: 1 Group: 0 Retention: 44:00 RIC: 977062 Masses: 43-433
Plotted: 1 to 5280 Range: 1 to 5280 100% = 12608042



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**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:	Semi-volatiles 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 in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
A409096	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

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Attachment 23.14
Figure 4
Analytical Run Logbook Example



Instrument Settings		Acquisition Information		Temperature Program				Miscellaneous
Multiplier: 1550	Scan Range: 40-450 amu	GC	Temp. (C)	Rate (C/min)	Time (min)	Total (min)	New Column Date: 4/6/14	
AGC 7000	Seconds/Scan: 0.500						New Septa: Yes (No)	
Interface Temp: 250C	Filament Delay: 3.5 min.	Step #1	45	1.5	1.5	Working Std. #: 54-33-9		
Source Temp: 190C	Threshold: 2 counts	Step #2	50	10	15.5	Int Std. #: 54-31-5		
Head Pressure: 8 psi	Mass Defect: 0	Step #3	315	2	4.6	Tune Std. #: 54-23-8		
	Background Mass: 35 amu	Step #4	315	0.0	16.4	Inj. Vol: 1 ul.		
	Curve Date: 11/30/14	Inject.	300	0.0	0.5	DDT Area 198: 993078		
		Inject.	300	100	2.55	DDT Breakdown: 0%		
		Inject.	300	0.0	15			

Sample / File ID	Client	Inj Time	Dil	M. Matrix	Function	Method	Notes
DFT 04/23C		8:47		QC	DFTPP	8270	Passed
5L04/23A		9:21			BNA		Passed
BLK04/23A		10:10		QC			
358328		11:12		WTR			
358329		11:14					
358330		15:05					
358331		15:45					
358332		14:25					
358333		15:09					
358334		15:49					
358335		16:24					
358336		17:09	STR	WTR	BNA	8270	

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.

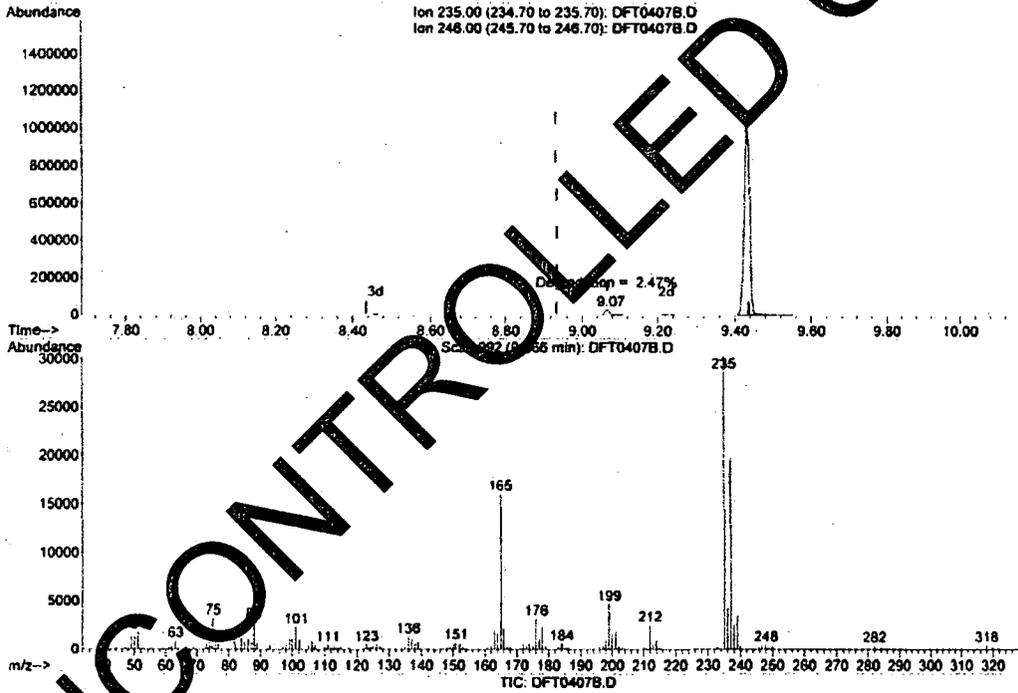
SOP Name: Base/Neutral/Acid Compounds by
Gas Chromatography/Mass Spectrometry
EPA Method 625, SW-846 Method 8270C
SOP Number: **GR-04-103** page 49 of 49

Revision Number: 5.8
Date Revised: 6/22/12
Date Initiated: 12/9/98

Attachment 23.16
4,4'-DDT Degradation Result Example

Data Path : C:\MSDCHEM\1\DATA\04-07-11_195\
Data File : DFT0407B.D
Acq On : 7 Apr 2011 10:09
Operator : DMC
Sample : DFT0407B
Misc :
ALS Vial : 2 Sample Multiplier: 1

Quant Time: Apr 07 10:26:55 2011
Quant Method : C:\MSDCHEM\1\METHODS\4DDT.M
Quant Title : 4-CHLOROPHENOL
QLast Update : Tue Mar 08 08:42:29 2011
Response via : Initial Calibration



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4,4'-DDT
9.07min (+0.130) 1.38ug/l
response 27722

Ion	Exp%	Act%
235.00	100	100
246.00	0.00	0.00
0.00	0.00	0.00
0.00	0.00	0.00



STANDARD OPERATING PROCEDURE

Dissolved Methane, Ethane and Ethene in Water by Headspace Equilibrium and Gas Chromatography

Newell SOP RSK-175

APPROVALS:

Area Supervisor: Jodi L. Blouw Date: 9-20-11
Jodi L. Blouw

QA Officer: Tom C. Boocher Date: 9-19-11
Tom C. Boocher

President: Douglas E. Kriscunas Date: 9-22-11
Douglas E. Kriscunas

Procedure Number: GR-03-130
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Date Revised: 9/19/11
Pages Revised: All

By: Andrea S. Colborn

Total Number of Pages: 22

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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 analysis is applicable to the determination of dissolved gases in non-saline groundwater by headspace equilibrium, at parts-per-million levels. Measurement of dissolved gases in water is used to monitor active bioremediation and natural attenuation.
- 1.2 This procedure is restricted to use by or under the supervision of analysts experienced in headspace and gas chromatography analysis. A demonstration of capability study must be performed by all analysts, before processing samples.

2.0 PRINCIPLE METHOD REFERENCES

- 2.1 Newell, B.S., RSKSOP-175, *Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using a GC headspace Equilibration Technique*, Revision 0, August 11, 1994
- 2.1.1 A modification to this procedure is that injection volumes used are 200 μL instead of 300 μL .
- 2.2 USEPA, Region 1, *Technical Guidance for the Natural Attenuation Indicators: Methane, Ethane, and Ethene*, Revision 1, 02/21/02

3.0 SUMMARY OF PROCEDURE

- 3.1 Water samples are preserved and collected in 40 mL VOA vials with PTFE-faced septum-lined caps, without headspace. Headspace is introduced at the laboratory by replacing a specified sample volume with ultra-high purity helium.
- 3.2 Water and headspace equilibration is performed by shaking each vial, for at least 5 minutes. A measured headspace volume is then removed and injected onto a gas chromatograph column. Target gases are separated on the column and detected by flame ionization (FID), for quantitation against a standard calibration.
- 3.3 Target gas concentration is calculated using Henry's Law constants (at the sample temperature), the quantitated headspace concentration, vial volume, and the sample temperature. The concentration of gas in the liquid is proportional to the partial pressure of the gas above the liquid.

4.0 PARAMETER OR COMPOUND LIST

Analyte	Linear Range (ug/L)	Reporting Limit (ug/L)	CAS Number	Molecular Weight (g/mole)
Methane	0-50	5.0	74-82-8	16
Ethene	0-50	5.0	74-85-1	28
Ethane	0-50	5.0	74-84-0	30

5.0 REFERENCED SOPs

- 5.1 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, 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

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

- 6.1 Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through vial septa, during shipment and storage. A field blank must be prepared from analyte-free reagent water and carried through all sampling and handling steps, to verify contamination by diffusion has not occurred.
- 6.2 Carryover can occur whenever low-level samples are sequentially analyzed after a high-level sample. To minimize carryover, sample syringes must be rinsed between gas chromatograph injections with helium gas. When a highly concentrated sample is analyzed, it must be followed by analysis of a syringe blank.
- 6.3 Before processing samples, the analyst must demonstrate an interference-free analytical system by analysis of an organic gas-free water blank. The blank analysis must be performed each day samples are analyzed. If interferences are detected at or above the reporting limit, the problem must be resolved before sample analysis begins.
- 6.4 Methane is a very common contaminant and occurs naturally in the atmosphere. Automobile exhaust also contains high levels of target gases. Care must be exercised to prevent contamination during transport and the field blank used to monitor contamination levels.
- 6.5 Moisture can interfere with low-level analysis. If a problem, moisture interference can be minimized by injecting through a calcium sulfate moisture trap.

7.0 SAFETY PRECAUTIONS

- 7.1 Wear a laboratory coat and approved safety glasses while in the 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.
- 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 exposure 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 Waste samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-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 Collect samples in duplicate pre-preserved 40 mL VOA vials.
- 8.1.1 Use hydrochloric acid (HCl) as the preservative.
- 8.1.2 Fill each vial by gently pouring down the inside of the glass without agitation, to just before overflowing then cap with the PTFE septum facing inward. Do not let the vial overflow.
- 8.1.3 Do not fill in a way that air bubbles pass through the sample. If headspace or air bubbles are trapped in the sample after a vial is sealed, repeat the sampling with a fresh vial.
- 8.2 Store collected samples at 0 - 6° C until analysis. Sample storage must be free of organic vapors.
- 8.3 Analyze samples within 14 days of collection. Samples not analyzed within this holding time must be narrated and qualified as estimated.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Glassware and Materials

- 9.1.1 Gas sampling bulbs for working standards, glass, with PTFE stoppers and a septum interface, 250 mL and 140 mL

Note: Verify the gas sampling bulb volume annually. Volume must be within ± 1 mL of 250 or 140 mL. If not, replace the bulb or use based on the actual volume measured.

- 9.1.2 Gas-tight syringe, with side-port needle, glass, Hamilton or equivalent, 500 μ L, 50 μ L, 10 μ L,
- 9.1.3 Screw-cap VOA vials, 40 mL, with PTFE-faced septa

- 9.1.4 Gas-tight syringe, with side-port needle, glass, Hamilton or equivalent, 5.0 mL
- 9.1.5 Gas standards at 10,000 uL/L (ppmv)
- 9.1.6 Helium, ultra-high purity or equivalent
- 9.1.7 Mechanical shaker, Burrell wrist-action shaker, model 75
- 9.1.8 Three-finger clamps
- 9.1.9 Ring stand

9.2 Instrumentation

- 9.2.1 Turbochrome data acquisition software, version 4.1, PE Nelson

Note: Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

Note: Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

- 9.2.2 Varian 3400 gas chromatograph, equipped with an FID detector

- 9.2.2.1 Injector Temperature: 250° C

- 9.2.2.2 Detector Temperature: 280° C

- 9.2.2.3 Column Temperature: 45° C Isothermal

- 9.2.2.4 Hold Time: 3.0 min

- 9.2.2.5 Packed Column: Supelco 50/80 Porapak P, 12' x 1/8"

- 9.2.3 HP Agilent 5890 gas chromatograph, equipped with and FID detector

- 9.2.3.1 Injector Temperature: 250° C

- 9.2.3.2 Detector Temperature: 280° C

- 9.2.3.3 Column Temperature: 30° C Isothermal

- 9.2.3.4 Hold Time: 3.0 min

- 9.2.3.5 Packed Column: Supelco 80/100 Porapak P, 6' x 1/8"

- 9.2.4 Injection Volumes: 2.0 µL to 500 µL

10.0 ROUTINE PREVENTIVE MAINTENANCE

- 10.1 Change GC injection port septa as needed.
- 10.2 Run helium blanks to clean out the column if poor chromatography or interferences appear. If running blanks does not resolve the problem, corrective action is required up to and including column replacement, before sample analysis can begin.

11.0 CHEMICALS AND REAGENTS

- 11.1 Laboratory reagent water (gas-free)
- 11.2 Gas standard cylinders containing methane, ethene and ethane at 1% (10,000 uL/L), purchased commercially and certified to be NIST traceable

12.0 STANDARDS PREPARATION

12.1 Stock Standard

12.1.1 The 10,000 uL/L gas mixture is withdrawn directly from the purchased cylinder as follows:

12.1.1.1 Turn on the gas so a steady stream of bubbles is emerging from cylinder and into a bubble which is a tubing line connected to a T-connector with the tubing end in a beaker of water. The other end of the T-connector contains a septum port for syringe extraction of the gas.

12.1.1.2 When the gas flow is steady, insert a syringe in the septum port and withdraw the appropriate gas volume for injection into the 250 mL gas sampling bulb.

12.2 Working Standards

12.2.1 Label a 250 mL gas sampling bulb as 200 uL/L and fill it with helium. Quickly rotate the stopcock several times to relieve excess pressure. With a gas-tight syringe, draw out 5.0 mL of helium. Purge the helium in the syringe to the atmosphere. With the same syringe, withdraw 5.0 mL of 10,000 uL/L standard (Section 12.1.1) and inject into the 200 uL/L bulb.

12.2.2 Different volumes of the 200 uL/L standard are injected to develop the calibration based on a 200 μ L injection, as follows:

Volume Injected (μ L)	Concentration (uL/L)	Concentration (P_{gas})
500	500	0.0005
100	100	0.0001

Volume Injected (μL)	Concentration (uL/L)	Concentration (P _{gas})
50	50	0.00005
20	20	0.00002
10	10	0.00001
4.0	4.0	0.000004

13.0 SAMPLE PREPARATION

- 13.1 Remove samples from the refrigerator and bring to room temperature before continuing.
- 13.2 To generate sample vial headspace, invert a sample vial in a three-finger clamp attached to a ring stand. Insert a needle attached to a Luer-Lok syringe through the septum. Connect another needle to the two-stage regulator of helium with a length of PTFE tubing. Insert the helium needle into the vial and inject helium at 5 mL/min or less. The helium will force sample into the syringe. Pull the helium line from the sample just before 10% of the total sample volume is reached. Allow the pressure in the vial to equalize before recording the actual volume of sample removed.
- Note: Purge the PTFE tubing with helium before preparing a sample and do not turn the gas off until all samples are prepared.
- 13.3 Shake the sample vial for at least 5 minutes to equilibrate headspace and liquid phases. After shaking, extract 200 μL of headspace with a 500 μL gas-tight syringe. If an immediate injection can not be made, vials must be kept inverted until the headspace can be analyzed.
- 13.4 Insert the syringe needle into the septum far enough so the needle port is fully in the headspace before extracting. As soon as the headspace is extracted, inject into a gas chromatograph for analysis.

14.0 CALIBRATION PROCEDURES

- 14.1 Analyze a helium blank before calibrating the GC to determine if the system is clean. If contamination is observed, correct the problem before calibrating.
- 14.2 Construct an initial six-point calibration curve by injecting each standard volume (Section 12.2.2). Inject sequentially from the lowest to highest concentration. The lowest standard must have a signal to noise ratio greater than 5. Plot peak area against concentration as a decimal fraction relating to partial pressure (For example: 10 uL/L is 0.00001 as a decimal fraction). A linear regression coefficient of 0.995 or higher must be achieved. Use only linear regression to calibrate with. Refer to Attachment 21.3 for a methane calibration example.

14.2 Continuing Calibration

After initial calibration, analyze, calibration verification standards (CCV). A CCV is a 100 uL/L standard (0.0001 as a decimal fraction), which must be analyzed initially then at a frequency of

once every 10 samples. CCV recovery must fall within 85 – 115%. If a target gas falls outside these acceptance limits, repeat the CCV analysis. If recovery is still unacceptable, correct the problem and rerun all samples analyzed since the last acceptable CCV.

15.0 ANALYTICAL PROCEDURE

- 15.1 After GC calibration, prepare samples by introducing headspace. After preparing, analyze immediately or keep vials inverted until the injection can be made. Use a 500 μL gas-tight syringe to inject 200 μL of headspace, as described in Section 13.0.
- 15.2 If a peak response exceeds the calibration range, inject a lesser volume of headspace. If less than 2 μL is required to put response within the calibration range, prepare a headspace dilution by extracting headspace from the sample and injecting into a gas sampling tube filled with helium.
- 15.3 Once sample analysis is complete and the final CCV is acceptable, take the temperature of the first sample prepared. This is the temperature (in Kelvin) that will be used in the calculations.

16.0 CALCULATIONS AND DATA HANDLING

- 16.1 Dissolved gas calculations involve several steps, as follows:

- 16.1.1 From sample analysis, an area count is obtained. Using area count and the standard curve, determine sample vial partial gas pressure.

NOTE: To develop the calibration regression, plot area count on the x-axis against the gas standard decimal fraction concentration on the y-axis (For example: 10 $\mu\text{L/L}$ is 0.00001 on the curve). The decimal fraction concentrations are unitless.

NOTE: In these calculations total pressure is assumed equal to one atmosphere, as follows:

$$\frac{P_{\text{gas}}}{P_{\text{total}}} = P_{\text{gas}}$$

The linear regression conforms to partial pressure based on the assumption, as follows:

$$P_{\text{gas}} = m(\text{area count}) + b$$

Where

P_{gas} = Partial pressure of the target gas (decimal fraction concentration)

m = Slope of the calibration line

b = Y-intercept of the calibration line

- 16.1.2 Calculate target gas concentration in the sample vial headspace phase, as follows:

$$\text{Concentration in headspace (mg/L)} = \frac{\text{MW g} \cdot \text{mole} \cdot 273 \text{ K} \cdot 1000 \text{ mg}}{\text{Mole} \cdot 22.4 \text{ L} \cdot T \text{ K} \cdot \text{g}} \cdot \frac{\text{mL}_{\text{headspace}} \cdot P_{\text{gas}}}{\text{mL}_{\text{water}}}$$

Where:

MW = Molecular weight of the target gas

T = Sample temperature, in Kelvins

mL_{headspace} = Sample vial headspace volume, in mL

mL_{water} = Sample vial water volume, in mL (calculate by subtracting headspace volume from total vial volume)

16.1.3 Calculate target gas concentration in the sample vial liquid phase, as follows:

$$\text{Concentration in Water Phase (mg/L)} = \frac{P_{\text{gas}} \cdot 55.5 \text{ mole} \cdot \text{MW g} \cdot 1000 \text{ mg}}{H_{\text{gas @ T}} \cdot \text{L} \cdot \text{mole} \cdot \text{g}}$$

Where:

H_{gas @ T} = Henry's law constant for target gas at the sample temperature

16.1.4 Calculate total target gas concentration, as follows:

TC = Concentration in headspace phase (mg/L) + Concentration in water phase (mg/L)

Where:

TC = Concentration of dissolved target gas in the sample, in mg/L

16.2 Example Calculation

16.2.1 Methane is used in the following example calculation. Parameters for the example are as follows:

Headspace Area Count	=	978264
Method Blank Area Count	=	2766
Henry's Law Constant	=	4.13E+04 @ 25° C
Sample Temperature	=	25° C (298.15 K)
Vial Volume	=	60 mL
Headspace Volume	=	6.0 mL
Molecular Weight	=	16 g/mole

16.2.2 Let the calibration curve be as follows:

$$P_{\text{gas}} = 1.814\text{E-}09(\text{area count}) - 6.716\text{E-}06$$

$$P_{\text{gas}} = 1.814\text{E-}09(978264 - 2766) - 6.716\text{E-}06$$

$$P_{\text{gas}} = 0.0018$$

16.2.3 Calculate target gas concentration in the sample vial headspace phase, as follows:

$$\text{Headspace Concentration (mg/L)} = \frac{16 \text{ g} \mid \text{mole} \mid 273 \text{ K} \mid 1000 \text{ mg} \mid 6 \text{ mL} \mid 0.0018}{\text{mole} \mid 22.4 \text{ L} \mid 298 \text{ K} \mid \text{g} \mid (60.6) \text{ mL}}$$

$$\text{Headspace Concentration (mg/L)} = 0.131 \text{ mg/L}$$

16.2.4 Calculate target gas concentration in the sample vial liquid phase, as follows:

$$\text{Concentration in Water Phase (mg/L)} = \frac{0.0018 \mid 55.5 \text{ mole} \mid 16 \text{ g} \mid 1000 \text{ mg}}{4.13 \times 10^4 \mid \text{L} \mid \text{mole} \mid \text{g}}$$

$$\text{Concentration in Water Phase (mg/L)} = 0.39 \text{ mg/L}$$

16.2.5 Calculate total target gas concentration, as follows:

$$\text{TC} = \text{Concentration in headspace phase (mg/L)} + \text{Concentration in water phase (mg/L)}$$

$$\text{TC} = (0.131 + 0.39) \text{ mg/L} = 0.521 \text{ mg/L methane}$$

16.3 A spreadsheet is available on the library drive of the laboratory intranet which performs all relevant calculations upon entering the area count. Refer to Attachment 21.1 for an example spreadsheet calculation.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analyst running samples are responsible for data quality and for filling in documentation correctly. It is important to document analysis by correctly filling in, handing in and filing paperwork. This is required for quality control and to provide clients with defensible data.

17.2 LIMS Reporting

17.2.1 When an analyst finishes running a sample batch, data must be input to LIMS (Element™). Data must be entered completely to ensure that results are reported correctly and data is associated with the right quality control batch. Dilution factors need added, so reporting limits are raised accordingly.

17.2.2 A Method Preparation Blank (BLK) must be run for each quality control batch, for each 24-hour shift. It is important to remember that a Blank Spike (BS) cannot be entered without also entering the associated BLK.

17.2.3 If internal chain-of-custody (CoC) is required, it is important that the CoC form be filled in and archived correctly.

17.2.4 All data hardcopy (including CoC forms) must be archived appropriately.

17.3 Laboratory Required Paperwork

17.3.1 All run, maintenance and standards logbooks (if used) must be filled in completely and accurately. Corrections must be made with a lineout, not a whiteout. Blank lines in run logbooks must be Z'd out, dated and initialed. Refer to Attachment 21.7 for an instrument run logbook example.

17.3.2 All initial calibration and CCV runs must be archived in the correct binder or hardcopy box.

17.3.3 All hardcopy documentation and raw data must be archived. Give these data to Data Management who must record the date, time and contents handed in. Sample and quality control benchsheets are returned to the proper folder after data review and approval.

17.4 Rounding and significant figures is to be performed only on final quantitated results by Element™.

18.0 QUALITY ASSURANCE

18.1 Continuing Calibration Verifications (CCV)

18.1.1 A CCV consists of a 100 µL/L standard, which must be analyzed at a frequency of one per 10 samples. CCV recoveries must fall within 85 – 115% for acceptance. If a CCV analyte recovery is outside the acceptance range, analyze another CCV to confirm. If results are still unacceptable, locate and correct the problem before processing further samples. All samples processed since the last acceptable CCV must be re-analyzed or narrated as estimated.

18.2 Method Preparation Blanks (BLK)

18.2.1 For each 24-hour analysis period, analyze a method preparation blank (BLK) to monitor for background contamination. The BLK is a 40 mL vial, filled with laboratory reagent water, prepared and analyzed as a sample. All target gas levels in the BLK must be less than the reporting limit. If contamination is found, locate the problem and correct before further samples are processed. All samples with concentrations above the reporting limit and processed since the last acceptable BLK, need re-analyzed or narrated as estimated.

18.3 Blank Spikes (BS)

18.3.1 For every batch of twenty samples or less a blank spike must be analyzed. A blank spike is a 40 mL vial containing laboratory reagent water prepared as a sample and spiked with 200 µL (after headspace is already introduced) of the 1% gas standard straight from the cylinder. The blank spike must have an acceptable recovery for all

target analytes. If the BS does not have acceptable recoveries, all samples in the batch must be re-extracted or qualified.

18.4 Matrix Spikes/Matrix Spike Duplicates (MS/MSD)

18.4.1 For every batch of twenty samples or less an MS/MSD pair must be extracted and analyzed. An MS/MSD is prepared by spiking a sample that already has headspace introduced with 200 µL of 1% gas standard straight from the gas cylinder. If there is not enough vials to prepare an MS/MSD pair a blank or a duplicate is prepared and all samples in the associated batch are qualified.

18.5 Verify each new calibration with a second-source calibration verification standard (SCV). An SCV must be ± 25% of the expected value to be acceptable. The SCV must be acceptable to begin sample analysis. If not acceptable, locate and correct the problem then repeat the SCV successfully. Prepare new standards and re-calibrate when no instrument malfunction is indicated.

18.5 Take corrective action for quality control problems in accordance with TriMatrix SOP GR-03-101.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATIONS

19.1 Before the analysis of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a demonstration of capability study. While demonstrations of capability are not instrument-dependent, one is required on every instrument running samples, to demonstrate the instrument's ability to generate acceptable accuracy and precision.

19.2 Prepare four gas-free SCV spikes in reagent water for the demonstration of capability study, as follows:

19.2.1 Create headspace in the sample vial, as outlined in Section 13.2. Keeping the vial inverted, inject an aliquot (200 µL) of the secondary stock cylinder containing methane, ethane and ethene. Continue the preparation including every step in the process and analyze exactly like a field sample.

19.2.2 Calculate the SCV concentration for each gas as follows:

$$\text{Spike amount (ug)} = \text{Gas Density (ug/}\mu\text{L)} * \text{Gas Volume (}\mu\text{L)}$$

$$\text{Spike amount (ug)} = \frac{\text{MW g}}{\text{mole}} \left| \frac{\text{mole}}{22.4 \text{ L}} \right| \left| \frac{273 \text{ K}}{\text{T K}} \right| \left| \frac{\text{STD } \mu\text{L}}{\text{L}} \right| \left| \frac{V_{inj} \text{ mL}}{\text{L}} \right| \left| \frac{\text{L}}{1000 \text{ mL}} \right|$$

Where:

- STD = Concentration of the spiking standard, in uL/L (ppmv)
- Vinj = Injection volume of the spiking standard, in mL
- T = Temperature during the process, in K
- MW = Molecular weight of the target gas, in g/mole

$$\text{SCV Concentration (mg/L)} = \frac{\text{Spike amount (ug)}}{V_{\text{water}} \text{ mL}}$$

Where:

V_{water} = Water phase volume, in mL

- 19.3 Analyze the four spikes following every step in the procedure.
- 19.4 Input to the IDC spreadsheet to calculate average recovery and relative standard deviation for each gas.
- 19.5 Results must be within the quality control limits associated with the SCV in Element™. If results are acceptable, the demonstration of capability study passes. The analyst and instrument are authorized to process samples.
- 19.6 If any gas fails, locate and correct the problem then repeat the study for the failed gas. Repeated failure indicates a general problem with the procedure and/or techniques used. If this occurs, locate problem, correct the procedure and/or techniques used then repeat the demonstration of capability study successfully. Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed.
- 19.7 A demonstration of capability study is required for each analyst annually.
- 19.8 A method detection limit (MDL) study in accordance with TriMatrix SOP GR-10-125 is also required annually.
- 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.
- 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 of material required.
- 18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal.

SOP Name: Dissolved Methane, Ethane, and Ethene in Water
by Headspace Equilibrium and Gas Chromatography
Newell SOP RSK-175
SOP Number: **GR-03-130**

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Revision Number: 0.5

Date Revised: 9/19/11
Date Initiated: 3/31/00

20.0 REFERENCES

20.1 Newell, B.S., RSKSOP-175, *Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using a GC headspace Equilibration Technique*, Revision 0, August 11, 1994

2.1.1 A modification to this procedure is that injection volumes used are 200 μL instead of 300 μL .

20.2 USEPA, Region 1, *Technical Guidance for the Natural Attenuation Indicators: Methane, Ethane, and Ethene*, Revision 1, 02/21/02

21.0 ATTACHMENTS/APPENDICES

21.1 Example Spreadsheet for Sample Calculations

21.2 Standards Logbook Example

21.3 Calibration Curve for Methane Example

21.4 Chromatogram Example

21.5 Preparation Batch Report Example

21.6 Data Review Report Example

21.7 Instrument Run Log Example

21.8 Method Detection Limit Study Example

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Attachment 21.1
Example Spreadsheet for Sample Calculations

Dissolved Gases in Ground Water
METHANE

Initial Cal ID:
METH_CRA.mth

Blank Area: 2319.00

AREA (x)	CONC. (y)
2048.00	0.000004
4245.00	0.000010
8894.00	0.000020
24284.00	0.000050
47083.60	0.000100
224316.68	0.000500
Slope:	2.23255E-09
Y Intercept:	-1.67258E-06
R:	0.999923

Analyte: **METHANE**
Molecular Weight (g/mol): 16
Henry's Law Constant: 41300
g-moles in 1 L. water: 55.5
Sample Temperature(K): 298
Density Correction Factor: 0.654
Bottle Volume(ml): 44
Headspace Volume(ml): 4.4

Sample	Sample Area Count	Analyte in Original Sample (ug/L)	Dilution Factor	Final Conc. (ng/L)	Partial Pressure	Equilibrium Mole Fraction	Moles of Gas	Saturation Conc. of Gas (mg/L)	Analyte in Head-space (mL)	Analyte in Liquid Phase (mg/L)
358388 20X	89074.00	18.58	20	271.59	0.0019719	4.77E-09	2.64988E-07	0.004239813	8.68E-04	0.01434
358389	157152.00	32.90	1	32.90	0.000349177	8.45E-09	4.69233E-07	0.007507729	1.54E-03	0.02539
358390 20X	85716.00	17.87	20	357.41	0.000189693	4.59E-09	2.54914E-07	0.004078621	8.35E-04	0.01379
358391 2X	138031.00	28.87	2	57.75	0.000306488	7.42E-09	4.11867E-07	0.006589873	1.35E-03	0.02228
358392 2X	142859.00	29.40	2	58.80	0.00031209	7.56E-09	4.19394E-07	0.006710311	1.37E-03	0.02269
358393	3290.00	0.85	1	0.05	4.95226E-07	1.20E-11	6.65497E-10	1.0648E-05	2.18E-06	0.00004
358499	8290.36	1.10	1	1.10	1.16588E-05	2.82E-10	1.56674E-08	0.000250678	5.13E-05	0.00085
358499DUP	5892.00	0.59	1	0.59	6.30432E-06	1.53E-10	8.47191E-09	0.00013555	2.77E-05	0.00046
358500 2X	165060.50	34.07	2	68.14	0.000361656	8.76E-09	4.86002E-07	0.007776039	1.59E-03	0.02629
358501 10X	10723.00	21.97	10	219.69	0.000233201	5.65E-09	3.13381E-07	0.005014094	1.03E-03	0.01696
358502 2X	14025.00	28.97	2	57.94	0.000307527	7.45E-09	4.13262E-07	0.006612194	1.35E-03	0.02236
358503 2X	12467.50	38.57	2	77.15	0.000409448	9.91E-09	5.50227E-07	0.008803628	1.80E-03	0.02977
358504 2X	13981.00	27.88	2	55.76	0.000295966	7.17E-09	3.97727E-07	0.006363637	1.30E-03	0.02152
358504DUP	12381.00	25.40	2	50.79	0.000269578	6.53E-09	3.62265E-07	0.005796247	1.19E-03	0.01960
358505 10X	99607.00	20.30	10	203.04	0.000215528	5.22E-09	2.89632E-07	0.004634106	9.48E-04	0.01567
358506	3376.57	0.06	1	0.06	6.88497E-07	1.67E-11	9.2522E-10	1.48035E-05	3.03E-06	0.00005
358507 10X	96972.00	19.75	10	197.50	0.000209645	5.08E-09	2.81726E-07	0.004507619	9.22E-04	0.01524
358508	3297.00	0.05	1	0.05	5.10853E-07	1.24E-11	6.86498E-10	1.0984E-05	2.25E-06	0.00004

SOP Name: Dissolved Methane, Ethane, and Ethene in Water
 by Headspace Equilibrium and Gas Chromatography
 Newell SOP RSK-175
 SOP Number: **GR-03-130**

Revision Number: 0.5

 Date Revised: 9/19/11
 Date Initiated: 3/31/00

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Attachment 21.2
Standards Logbook Example

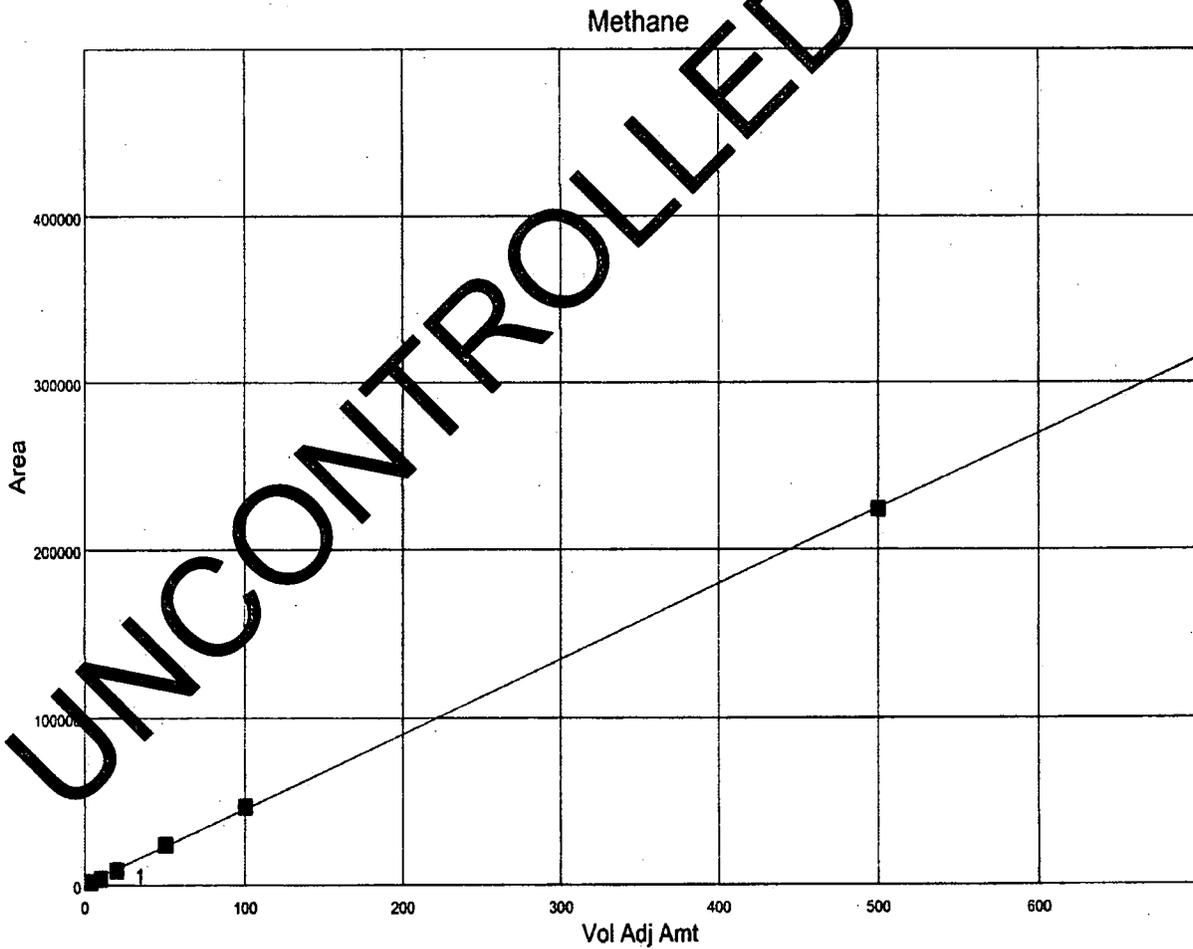

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/Volume	Solvent Used/ Lot #	Final Volume	Final Concentration	Prepared By	Date Made or Opened	Date Expires	Math Check By
1	GC3.34-1	Diesel #2 w/ Triolein	GL323-1	-	2/19/05	1000 µg/ml	9 ml	MeCl	10 ml	1000 µg/ml	JCM	9/7/04	12/1/04	
2	GC3. -2		GL3 PL4.9-2	-	2/19/05	500 µg/ml	1 ml	↓	↓	500 µg/ml	↓	↓	↓	
3	GC3.34-3	1242	1242	Absolute 90126 lot # 010903	10/5/05		5 µl	Hex	25 ml	0.80 µg/ml	JCM	10/26/04	4/5/05	
4	GC3. -4		PL4.10-14	-	4/5/05		5 ml	↓	↓	0.20 µg/ml	↓	↓	↓	
5	GC3.34-5	1254	1254	Absolute 70020 lot # 020100	11/20/04		5 µl	↓	↓	0.80 µg/ml	↓	↓	11/20/04	
6	GC3. -6		PL4.10-14	-	5/05		5 ml	↓	↓	0.20 µg/ml	↓	↓	↓	
7	GC3.34-7	Chlordane	CHLORDANE	Ultra lot # 5081	1/2/05	1 µg/ml	50 µl	Hexane	50 ml	0.100 µg/ml	SSPS	10/26/04	2/23/05	
8	GC3. -8		PL4.10-5 (Sur)	-	9/27/05	1 µg/ml	5 ml	↓	↓	↓	SSPS	↓	↓	
9	GC3.34-9	AP II i-high	Isodrin	Absolute 70015 lot # 011320	11/7/05	100 µg/ml	5 µl	Hexane	25 ml	0.2 µg/ml	MP	10/25/04	1/19/05	
10	GC3. -10		Chlordane	Absolute 70020 lot # 020100	5/11/05		50 µl	↓	↓	2.0 µg/ml	↓	↓	↓	
11	GC3. -11		Chlordane	Absolute 70020 lot # 020100	5/11/05		50 µl	↓	↓	2.0 µg/ml	↓	↓	↓	
12	GC3. -12		Chlordane	Absolute 70020 lot # 020100	5/11/05		50 µl	↓	↓	2.0 µg/ml	↓	↓	↓	
13	GC3. -13		TCM + DCB	-		1.0 ppm	5 ml	↓	↓	0.2 µg/ml	↓	↓	↓	
14	GC3.34-14	DCB	TCM + DCB	Ultra Pm gas lot # 4-4622	1/2/05	100 ppm	8 µl	↓	50 ml	6.16 ppm	↓	11/2/04	1/7/05	
15	GC3. -15		TCM + DCB	-		1.0 ppm	8 ml	↓	↓	6.16 ppm	↓	↓	↓	
16	GC3.34-16	10160	10160	Absolute 70015 lot # 011323	11/1/05	1000 ppm	40 µl	Hex	25 ml	1.6 ppm	JCM	11/4/04	5/4/05	
17	GC3. -17		1260	Absolute 70021 lot # 051600	5/16/05		↓	↓	↓	1.6 ppm	↓	↓	↓	
18	GC3. -18		TCM + DCB	PL4.11-9	5/11/05	1.0 ppm	5.0 ml	↓	↓	0.20 ppm	↓	↓	↓	

Attachment 21.3
Calibration Curve for Methane Example

Fit Analysis Output For Method File: "C:\TC4\159\RSK42304.MTH"
Component Name: "Methane"
Date: 11/22/04 Time: 10:37

Curve Parameters:

Curve #1 : 1st Order
Weighting Factor = 1.0 (No Weighting) $r^2 = 0.999846$
Calibration Curve = $(757.021844) + (447.849633)X$



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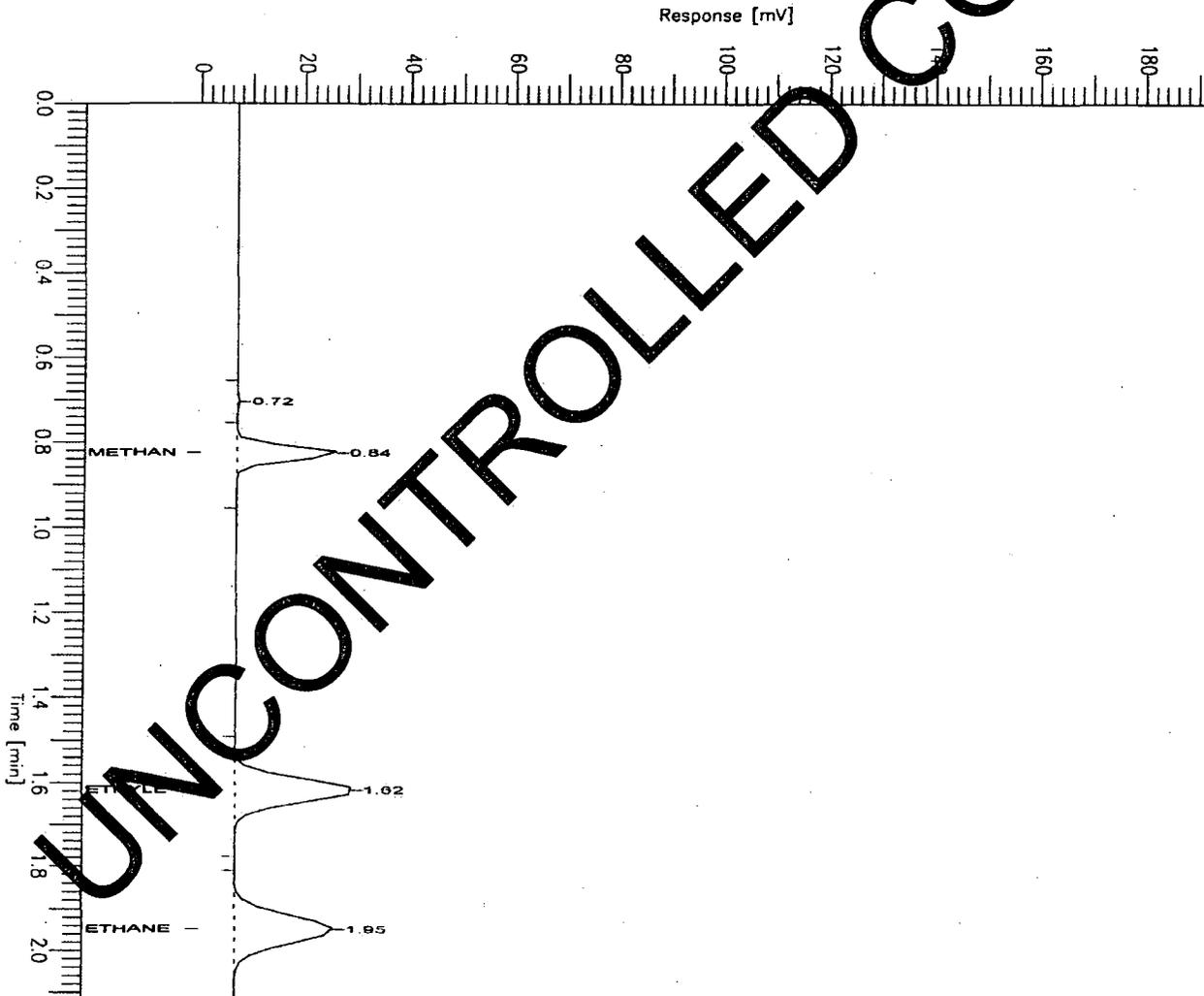
Date Revised: 9/19/11

Date Initiated: 3/31/00

Attachment 21.4
Chromatogram Example

Chromatogram

Sample Name :
FileName : C:\TC4\159\B57_070.RAW
Method : RSK42304.MTH
Start Time : 0.00 min
Scale Factor : 0.0
Sample #: CCV/LFB
Date : 11/22/04 10:43
Time of Injection: 11/3/04 13:00
Low Point : 0.00 mV
Plot Scale: 200.0 mV
Page 1 of 1
High Point : 200.00 mV



SOP Name: Dissolved Methane, Ethane, and Ethene in Water
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Attachment 21.5
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH **0707324** Page 1 of 1

Date: 6/16/2007 4:21:20PM

Semivolatiles GC, Water, Direct Injection
 (No Surrogate)

Batch Comments: (none)

<u>Work Order</u>	<u>Analysis</u>	<u>Work Order</u>	<u>Analysis</u>	<u>Work Order</u>	<u>Analysis</u>
0706375	RSK-175	0706397	RSK-175		

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client QC Type	Extraction Comments
0707324-BLK1		Jun-29-07 12:33	JLW	1	1					BLANK	
0707324-DUP1		Jun-29-07 12:33	JLW	1	1		0706397-10			DUPLICATE	
0707324-BS1		Jun-29-07 12:33	JLW	1	1			6100100	10	LC#	
0706375-01	C	Jun-29-07 12:33	JLW	1	1						
0706375-02	C	Jun-29-07 12:33	JLW	1	1						
0706375-03	C	Jun-29-07 12:33	JLW	1	1						
0706375-04	C	Jun-29-07 12:33	JLW	1	1						
0706375-05	C	Jun-29-07 12:33	JLW	1	1						
0706375-06	C	Jun-29-07 12:33	JLW	1	1						
0706397-01	C	Jun-29-07 12:33	JLW	1	1						
0706397-02	C	Jun-29-07 12:33	JLW	1	1						
0706397-03	C	Jun-29-07 12:33	JLW	1	1						
0706397-04	C	Jun-29-07 12:33	JLW	1	1						
0706397-05	C	Jun-29-07 12:33	JLW	1	1						
0706397-06	C	Jun-29-07 12:33	JLW	1	1						
0706397-07	C	Jun-29-07 12:33	JLW	1	1						
0706397-08	C	Jun-29-07 12:33	JLW	1	1						
0706397-09	C	Jun-29-07 12:33	JLW	1	1						
0706397-10	C	Jun-29-07 12:33	JLW	1	1						

Comments	Analyst Initials
----------	------------------

Attachment 21.6
Data Review Report Example

Sequence = 7062967 Page 1 of 1

on 7/6/2007 at 4:35

SampleID	Analysis	Result	Qualifier	Units	Diln	FResult	FUnits	MDL	LMRL	FMRL	Recovery	RPD	Analzyed
0707324-BS1	RSK-175	17.6		ug/L	1	17.6	ug/L	0.147	1	1.0	96		6/29/2007 1:27
0707324-BS1	RSK-175	16.47		ug/L	1	16.5	ug/L	0.167	1	1.0	100		6/29/2007 1:27
0707324-BS1	RSK-175	9.08		ug/L	1	9.08	ug/L	0.0785	0.5	0.50	96		6/29/2007 1:27
0707324-BLK1	RSK-175	0		ug/L	1	0.00	ug/L	0.147	1	1.0			6/29/2007 1:34
0707324-BLK1	RSK-175	0		ug/L	1	0.00	ug/L	0.0785	0.5	0.50			6/29/2007 1:34
0707324-BLK1	RSK-175	0		ug/L	1	0.00	ug/L	0.167	1	1.0			6/29/2007 1:34
0706375-01	RSK-175	4.13		ug/L	1	4.1	ug/L	0.0785		0.50			6/29/2007 1:38
0706375-01	RSK-175	0		ug/L	1	0.0	ug/L	0.167	1	1.0			6/29/2007 1:38
0706375-01	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:38
0706375-02	RSK-175	45.88		ug/L	1	46	ug/L	0.0785	0.5	0.50			6/29/2007 1:42
0706375-02	RSK-175	2.82		ug/L	1	2.8	ug/L	0.167	1	1.0			6/29/2007 1:42
0706375-02	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:42
0706375-03	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:45
0706375-03	RSK-175	1.08		ug/L	1	1.1	ug/L	0.167	1	1.0			6/29/2007 1:45
0706375-03	RSK-175	5.75		ug/L	1	5.8	ug/L	0.0785	0.5	0.50			6/29/2007 1:45
0706375-04	RSK-175	2.22		ug/L	1	2.2	ug/L	0.0785	0.5	0.50			6/29/2007 1:49
0706375-04	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:49
0706375-04	RSK-175	0.94		ug/L	1	0.94	ug/L	0.167	1	1.0			6/29/2007 1:49
0706375-05	RSK-175	4.45		ug/L	1	4.4	ug/L	0.0785	0.5	0.50			6/29/2007 1:52
0706375-05	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:52
0706375-05	RSK-175	2.21		ug/L	1	2.3	ug/L	0.167	1	1.0			6/29/2007 1:52
0706375-06	RSK-175	0.06		ug/L	1	0.060	ug/L	0.167	1	1.0			6/29/2007 1:56
0706375-06	RSK-175	16.76		ug/L	1	17	ug/L	0.0785	0.5	0.50			6/29/2007 1:56
0706375-06	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:56
0706397-10	RSK-175	3.92		ug/L	1	3.9	ug/L	0.0785	0.5	0.50			6/29/2007 2:37
0706397-10	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 2:37
0706397-10	RSK-175	1.31		ug/L	1	1.3	ug/L	0.167	1	1.0			6/29/2007 2:37
0707324-DUP1	RSK-175	0		ug/L	1	0.00	ug/L	0.147	1	1.0			6/29/2007 2:40
0707324-DUP1	RSK-175	1.29		ug/L	1	1.29	ug/L	0.167	1	1.0		2	6/29/2007 2:40
0707324-DUP1	RSK-175	3.93		ug/L	1	3.93	ug/L	0.0785	0.5	0.50		0.3	6/29/2007 2:40

SOP Name: Dissolved Methane, Ethane, and Ethene in Water
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Date Initiated: 3/31/00

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Attachment 21.7
Instrument Run Logbook Example



Instrument ID: Varian 3400 #159		Date: 9/8/04	Sequence #: 856		Date Archived, Disk ID				
Instrument Settings/Injection Volume			Breakdown		Quantitation Information			Routine Maintenance Items	
Column Type: DB624 / Porapak	Curve Date:	GC	Program 1	Program 2	New Column Date:				
Injection Volume: 1 ul / 2 uL / 200 ul	Curve Type: Regression or Average CF	Initial: 45°	-	-	Column Shipped: Yes (No)				
Head Pressure: 20 psi	Quantitated By: Curve	Hold: 3min	-	-	New Injection Port Liner: Yes (No)				
Injector Temperature: 250° C		Final: -	-	-	New Septa: Yes (No)				
Detector Temperature: 280° C		Rate: -	-	-	New Syringe: Yes (No)				
Program Name:		Hold: -	-	-					
Analyst	Run ID	File ID	Injection Time	Method	Client	Matrix	Injection	ICV/CCV Check (85-115)	Sample Notes, Standard Numbers, Analytical Batch Information, etc.
JTM	Inst BLK	1	14:15	RSK42304		QC		PASS	
	CCV/LFB	2	14:19			QC		PASS	S-2132
	MPB	3	14:23			Wtr			
	369189	4	14:26						
	369189	5	14:47				20x		
	369190	6	14:50						
	369191	7	14:51						
	369192	8	14:53						
	369192	9	15:02				20x		
	369193	10	15:07						
	369194	11	15:10				20x		
	369194	12	15:16	JTM 9/8/04			20x	JTM 9/8/04	
	CCV	13	15:20			QC		PASS	S-2132
	369194dup	14	15:25		Merit	Wtr	20x		
	CCV	15	15:33						S-2132

SOP Name: Dissolved Methane, Ethane, and Ethene in Water
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Newell SOP RSK-175

Revision Number: 0.5

SOP Number: GR-03-130

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Date Revised: 9/19/11

Date Initiated: 3/31/00

**Attachment 21.8
Method Detection Limit Study Example**

SEMI-VOLATILE LABORATORY
INSTRUMENT NUMBER 159 2804 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
METHANE	RSK-175	1/28/2004	0.500	ug/L	0.160	0.090	0.120	0.130	0.090	0.060	0.130	0.111	22%	0.0334	0.105
ETHYLENE	RSK-175	1/28/2004	0.500	ug/L	0.470	0.560	0.510	0.470	0.520	0.580	0.480	0.513	103%	0.0439	0.138
ETHANE	RSK-175	1/28/2004	0.500	ug/L	0.500	0.590	0.540	0.500	0.500	0.620	0.530	0.549	110%	0.0449	0.141

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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 L. VanMale* Date: 1-12-12
Diane L. VanMale

QA Officer: *Tom C. Bochner* Date: 1-12-12
Tom C. Bochner

Laboratory President: *Douglas E. Kriscunas* Date: 1-12-2012
Douglas E. Kriscunas

Procedure Number: GR-04-104
Revision Number: 4.7

Date Initiated: 9/1/95
Effective Date: 1/25/12

Date Revised: 1/12/12
Pages Revised: All

By: Diane L. VanMale

Total Number of Pages: 43

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
_____	_____	_____
_____	_____	_____
_____	_____	_____

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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.
- 1.3 For analysis of nonaqueous matrices, refer to TriMatrix SOP GR-04-105.

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, latest revision*
- 2.3 *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 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 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 effuse 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. The storage blank is analyzed for an extensive list of volatile analytes. A TIC scan is also performed. If positive results are observed for any target analyte above the laboratory's minimum reporting limit, all samples stored concurrently in the CTU must be evaluated for possible contamination. All sample results within 5 times the level quantitated in the storage blank must be qualified as estimated.
- 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. Varying methanol can suppress analyte signal response, and can alter certain 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 Wear a laboratory coat and approved safety glasses while in the laboratory (safety glasses are required when entering data into a computer). 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.
- 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 exposure 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 Waste samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-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 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 vial with a septa screw-cap lid (PTFE face must be down), then cool to 0 - 6° 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 0 - 6° C.

8.1.1.4 Analysis of 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 Preferably, Method 624 samples may be collected with preservative as described above. Or, they may be collected with no preservative. In either case, samples must be cooled to 0 - 6° 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. The 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 0 - 6° 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 0 - 6° C.

8.2.1 Once received, TriMatrix must store samples at 0 - 6° 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 (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 0 - 6° 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/Encon Evolution (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 Centurion (Environmental Sample Technology) for water samples, Methanol preserved soil samples, and low-level soil samples.
- 9.2.3 Gas Chromatograph
- 9.2.3.1 Hewlett Packard Model 7890 . 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 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 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 5975C 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: 230° C

9.2.6 Data Acquisition:

9.2.6.1 The HP MSD data acquisition system is a DOS-based HP Chemstation equipped with EnviroScan environmental data analysis software. It is also capable of plotting EIPs and has a 120,000 compound NIST spectral library.

9.2.6.2 Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

9.2.6.3 Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Mass Spectrometer Maintenance

10.1.1 Mass Selective Detector (MSD) Maintenance Schedule:

10.1.1.1 Drain and replace rough pump oil every 6 months

10.1.1.2 Check diffusion pump oil annually and change if discolored or low

10.1.1.3 Clean ion source at least annually (may be needed more frequently as shown by detector performance)

10.1.1.4 Replace electron multiplier as needed (maximum voltage 3000)

10.1.1.5 Check calibration gas and refill as necessary

10.2 Purge and Trap Maintenance Schedule:

- 10.2.1 Before initial use, new traps must be conditioned for one hour at 260C.
- 10.2.2 Check and record the purge flow weekly
- 10.2.3 Empty waste bottles and fill rinse bottles daily
- 10.2.4 Check and record the concentrator pressures weekly
- 10.2.5 Check and if necessary fill internal/surrogate standard module as needed.
- 10.2.6 Clean the 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 by volume
- 11.5 Sodium bisulfate, NaHSO₄, ACS reagent grade or equivalent

12.0 STANDARDS PREPARATION

- 12.1 All laboratory standards must be recorded 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 recommendations, and are 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 14.7 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 or chloromethane will generally be the first of the gaseous compounds to fail.

12.2.2.3 Because 2-chloroethylvinyl 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 mini-inert vials at or below -10° C. 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 0.001 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 at 2.0 mg/mL in Methanol
- Mix B: Volatile Organic Compounds - Gases at 2.0 mg/mL in Methanol
- Mix C: Acrolein & Acrylonitrile at 2.0 mg/mL in Methanol
- Mix D: Ketone Mix at 2.0 mg/mL in Methanol
- Mix E: Custom Additions to Method 8260B at 2.0 mg/mL in Methanol
- Mix F: Dichlorofluoromethane at 1.0 mg/mL in Methanol

- Mix G: Internal Standard and Surrogate Standard Mix at 5.0 mg/mL in Methanol.

- Mix H: BFB Standard Mix at 2.0 mg/mL in Methanol.

12.3.4.2 The following standards are combined to make Stock Standard II.

- Mix A: Appendix IX Standard at 2.5 mg/mL in Methanol.
- Ethyl Methacrylate at 1.0 mg/mL in Methanol.
- Isopropanol at 1.0 mg/mL in Methanol.
- 2-Chloro-1,3-butadiene (chloroprene) at 1.0 mg/mL in Methanol.
- N-butyl acetate at 2.0 mg/mL in Methanol.
- Methyl Methacrylate at 2.0 mg/mL in Methanol.

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 μ L 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 μ L Mixes A, B, C, D, and E
- 100 μ L 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 610 μ L of methanol to the mini-inert vial. From the mixes specified in Section 12.3.4.2 add the following quantities to produce 1.0 mL of a 100 mg/L intermediate stock standard:

- 40 μ L Mix A
- 50 μ L 2-Chloro-1,3-butadiene
- 100 μ L Isopropanol
- 100 μ L Ethyl methacrylate
- 50 μ L Methyl methacrylate
- 50 μ L N-Butyl acetate

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 μ L of methanol to the mini-inert vial. Add 10 μ L 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. These standards are prepared as needed and not stored. Calibration verification standards are typically prepared at 40 ug/L for all except Appendix B 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 (μ L)
- 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 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 Defense must be spiked with 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 are prepared at the same concentration as the matrix spike.

12.7.2 The same analytes used in matrix spikes are 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 that must meet data quality objectives of the Department of Defense must be spiked with 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 and analyzed using the Centurion Autosampler

13.2.1 Samples analyzed on this autosampler 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 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 μ L of internal standard/surrogate mixture before purging. The Centurion is 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 23.6 or 23.7. 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 23.6 or 23.7, 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:

$$RF_{avg} = (RF_1 + RF_2 + RF_3 + \dots + RF_n)/n$$

where:

- RF_{avg} = Average calibration response factor
- RF₁ = Calibration response factor for standard 1
- RF₂ = Calibration response factor for standard 2
- RF₃ = 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. 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:

$$\text{Percent RSD} = \frac{\text{SD}}{\text{X}} \times 100$$

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 $\leq 15\%$. Six compounds are used as Calibration Check Compounds (CCCs): 1,1-dichloroethene, chloroform, 1,2-dichlorobenzene, toluene, ethyl benzene, vinyl chloride. These compounds **must** have a percent RSD of $\leq 30\%$. 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 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:

$$C_i = (A_i - b)/m$$

Where:

- C_i = Concentration in ug/mL
- A_i = Area ratio ($A_{\text{analyte}}/A_{\text{IS}}$)
- 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 data 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 $\pm 25\%$ 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 15.3). If an initial calibration has just been run, then the 40 ug/L standard from the calibration can be 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:

$$\text{Percent Drift} = [(C_c - C_1)/C_1] * 100$$

where:

C₁ = CCC concentration

C_c = Measured concentration using selected average RF or regression.

$$\text{Percent Difference} = [(RF_{ccc} - RF_{avg})/RF_{avg}] * 100$$

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 ±20%, 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.

14.7.4.4 For any analyte that does not meet the above criteria, the appropriate qualifier must be attached.

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 the 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 To prepare a working BFB standard solution, spike 25 µL into a 50 mL volumetric flask containing reagent water. Transfer the contents to a 40 mL sample vial.
- 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 give an acceptable BFB tune, then corrective action is required.
- 15.3.3.3 The first step is to reinject and reanalyze the same BFB standard.
- 15.3.3.4 Next, prepare a new BFB working solution, then inject and analyze.
- 15.3.3.5 The last step is to optimize the tuning acquisition parameters by tweaking manually or by performing a new autotune.
- 15.3.3.6 Once acquisition parameters are optimized, restart the tuning sequence.

15.4 Initial/Continuing Calibration

- 15.4.1 After a successful BFB analysis, an initial calibration curve or continuing calibration verification must be analyzed (Section 14.0). Initial and continuing calibrations for soil sample analysis must match sample methanol and 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 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 surrogates 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 40° C and held for 30 seconds. 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.

15.5.4 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 50 mL volumetric flask. Minimum a 1:50 dilution is done.

15.5.4.2 Transfer the diluted extract to a 40 mL vial and place in the autosampler, as in 15.5.1.

15.5.4.3 Report soil results on a dry weight basis. Report waste results on a wet weight basis.

15.5 Sample Desorption

15.6.1 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 is prepared 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 search 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 $\pm 20\%$. 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 estimates 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

$$\frac{A_x \times C_{is}}{A_{is} \times RF} \times DF = \text{ug/L}$$

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

$$\frac{\text{mg}}{\text{kg}} = \frac{A_x \times C_{is}}{A_{is} \times \text{RF}} \times \frac{5 \text{ mL}}{W \times \%S} \times \frac{L}{1000 \text{ mL}} \times \frac{\text{mg}}{1000 \text{ ug}} \times \frac{1000 \text{ g}}{\text{kg}}$$

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

$$\frac{\text{mg}}{\text{kg}} = \frac{A_x \times C_{is}}{A_{is} \times \text{RF}} \times \frac{\text{DF}}{\%S} \times \frac{V}{W} \times \frac{L}{1000 \text{ mL}} \times \frac{\text{mg}}{1000 \text{ ug}} \times \frac{1000 \text{ g}}{\text{kg}}$$

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-10-113.

17.3 If an integral 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 verification, 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.
- 17.6 With two exceptions, all spectra, whether designated by the analyst as a positive hit or a false positive, must be retained for peer review.
- 17.6.1 Exception #1: Peer review will not be required, nor will a hard copy of the spectra be retained, if the false positive is based on a poor retention time match (making spectral match irrelevant).
 - 17.6.2 Exception #2: Peer review will not be required, nor will a hard copy of the spectra be retained, for analytes detected below the reporting limit (when reporting of estimated results is not required).
- 17.6.3 All retained spectra must be initialized by the peer performing the review.

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 Verification (Section 14.6)

18.1.3 Continuing Calibration Verification (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. A BLK may 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 on 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:

$$\text{Upper Control Limit (UCL)} = p + 3s$$

$$\text{Lower Control Limit (LCL)} = p - 3s$$

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.

- 18.5.1.3 An LFB/BS may be from the same source as the calibration or from the second-source used to verify the initial 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 Calculate recoveries and compare to LIMS control limits. Stop analysis and correct the problem if recoveries are outside quality control limits.
 - 18.5.4.2 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 failed analytes must be qualified as estimated.
 - 18.5.4.3 If 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.
 - 18.5.4.4 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.
 - 18.5.4.5 Take corrective action in accordance with TriMatrix SOP GR-03-124.

18.6 Matrix Spikes (MS, MSD)

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Column Gas Chromatography/Mass Spectrometry
SW-846 Method 8260B, EPA Method 624
SOP Number: **GR-04-104** page 28 of 43

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Date Revised: 1/12/12
Date Initiated: 9/1/95

- 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 SPCCs and CCCs are not used. Instead response factors for every compound listed on the EPA 624 list must have 100% RSD for the calibration to be valid.
- 18.7.3 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, an appropriate qualifier will be attached to that analyte.
- 18.7.4 There are no criteria for internal standard areas or retention times in method 624. However, method 8260B criteria will be followed.
- 18.7.5 Every targeted analyte must be spiked in the MS/MSD, and LFB/BS. Not just the limited list in Section 18.5.2.
- 18.7.6 Acrolein and Acrylonitrile may only be screened by GC/MS. All positive results must be qualified in the report as estimated.
- 18.7.7 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 $\leq 20\%$.

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 all 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 all 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.

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- 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, latest revision*
- 22.3 *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

SOP Name: Volatile Organic Compounds by Purge and Trap Capillary
Column Gas Chromatography/Mass Spectrometry
SW-846 Method 8260B, EPA Method 624
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Date Initiated: 9/1/95

23.10 Attachment 23.10, Example Chromatogram, DB-VRX

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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	10	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	67-66-0	100	1	0.01	0.05
B	Chloromethane	74-87-3	100	1	0.01	0.05
A	2-Chlorotoluene	95-47-8	100	1	0.01	0.05
A	4-Chlorotoluene	106-43-3	100	1	0.01	0.05
E	Cyclohexane	118-97-7	100	10	0.01	0.25
A	Dibromochloromethane	74-48-1	100	1	0.01	0.05
A	1,2-Dibromo-3-chloroethane	95-12-8	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	Dichlorodibromomethane	75-71-8	100	1	0.01	0.05
A	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	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	0.1	0.05
E	Hexachloroethane	67-72-1	100	1	0.1	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	0.1	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	1	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	1	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	630-00-6	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	88-41-6	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-2,2,2-trifluoroethane	76-13-1	100	1	0.01	0.05
A	2,4-Trimethylbenzene	95-63-6	100	1	0.01	0.05
A	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	104-89-8	250	25	0.05	1.3
A	Ethanol	64-17-5	250	50	0.5	2.5
A	Ethyl acetate	78-17-6	250	10	0.5	2.5
Neat	Ethyl methacrylate	97-63-2	250	5	0.05	0.25
A	Hexachloroethane	87-72-1	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	Methyl cyclohexane	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

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

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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

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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.

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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	Dichlorodifluoromethane	85	87, 99	1
2	Chloromethane	50	52, 41	1
3	Vinyl Chloride	62	64, 47	1
4	Bromomethane	94	96, 93	1
5	Chloroethane	64	66, 41	1
6	Trichlorofluoromethane	101	103, 105	1
7	Dichlorofluoromethane	69	69, 47	1
8	Ethyl Ether	45	59, 73	1
9	Acrolein	53	56, 53	1
10	1,1-Dichloroethylene	96	61, 98	1
11	1,1,1-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	99	127, 131	2
54	1,2-Dibromoethane	105	107, 188	2
55	IS:d5-Chlorobenzene	72	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	o-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
	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	53	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	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	Tetrachlorohydrin	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 (µg/L)
Benzene	25.8-54.4
Bromodichloromethane	23.2-53.8
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=Detector result must be greater than zero.

SOP Name: Volatile Organic Compounds by Purge and Trap Capillary
 Column Gas Chromatography/Mass Spectrometry
 SW-846 Method 8260B, EPA Method 624
 SOP Number: **GR-04-104**

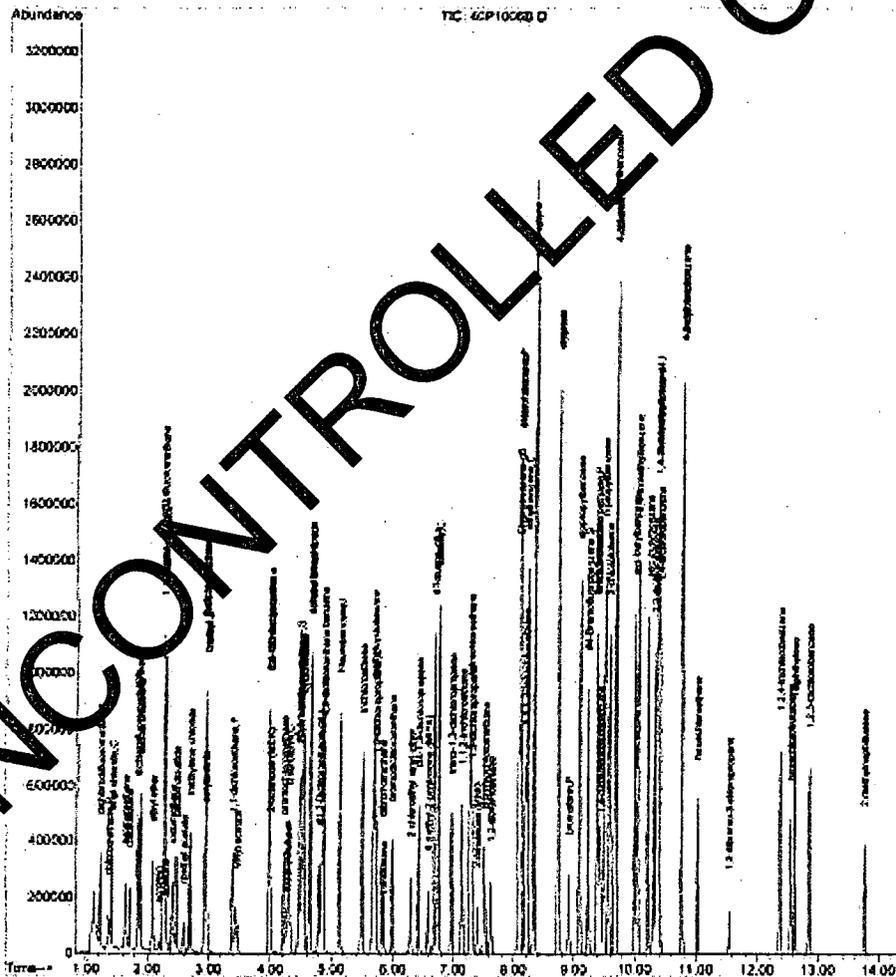
Revision Number: 4.7
 Date Revised: 1/12/12
 Date Initiated: 9/1/95

Attachment 23.9
Example Chromatogram, DB-624

Quantitation Report (Not Edited)

Data Path : C:\MSDCHEM\1\DATA\10-06-11\
 Last Name : 224
 Data File : 40P1006B.D
 Acq On : 6 Oct 2011 15:45
 Operator : DLV
 Sample : CALS
 Misc :
 ALS Vial : 7 Sample Multiplier: 1

Quant Time: Oct 06 15:59:26 2011
 Quant Method : C:\MSDCHEM\1\METHODS\8260B-808A.M
 Quant Title : 8260B/624/524.2 : 5030.wel : 5035a.wel
 Qlist Update : Thu Oct 06 11:46:37 2011
 Response via : Initial Calibration



8260B-B99.M Fri Oct 14 12:36:37 2011

Page: 3



STANDARD OPERATING PROCEDURE

Closed System Purge and Trap and Extraction for Volatile Organic Compounds

SW-846 Method 5035A

APPROVALS:

Area Supervisor: *Diane VanMale* Date: 8-7-12
 Diane L. VanMale

QA Officer: *Tom C. Booher* Date: 8-8-12
 Tom C. Booher

Laboratory President: *Douglas E. Kriscunas* Date: 8/6/2012
 Douglas E. Kriscunas

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By: Diane L. VanMale
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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 uses a hermetically-sealed 40 mL sample vial, the seal of which is never broken from the time of collection to the time of analysis. 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, surrogates and internal standards are automatically added by the autosampler 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 approximately 10 g of sample weighed in the field in a pre-weighted vial with a septum-sealed screw cap that contains 10 mL of methanol for extraction. Upon laboratory receipt, samples are shaken for two minutes and sonicated for 20 minutes. For analysis, 1.0 mL of the solvent extract is diluted into 50 mL laboratory reagent water. The solution is transferred to a new pre-cleaned VOA vial for analysis. Surrogates and internal standards are added by the autosampler immediately before purging and analysis in accordance with the appropriate analytical procedure.
- 3.2.2 The second 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. Samples are then shaken for two minutes and sonicated for 20 minutes. For analysis, 1.0 mL of the solvent extract is diluted into 50 mL laboratory reagent water. The solution is transferred to a new pre-cleaned VOA vial for analysis.

Surrogates and internal standards 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 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.

Note: This is the least desirable option as the sample contacts the atmosphere which may cause some volatile constituent losses.

- 3.3 High concentration oily waste procedure - Applicable to oily samples with VOA 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.

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 elevated 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 Wear a laboratory coat and approved safety glasses while in the laboratory (the only exception is when entering data at a computer terminal). 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.
- 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 exposure 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 Waste samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-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 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. Wear disposable gloves during the following preparation steps:

8.2.1 Prepare vials used in the collection of low concentration soil samples that are to be preserved in the field as follows:

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 must only be added after sample has been added. This will be done automatically by the autosampler just prior to analysis.

8.2.2 For low level soil samples that are to be preserved by freezing, follow step 8.2.1.1, then steps 8.2.1.3 through 8.2.1.6 above.

8.2.2.1 Preservation by freezing to less than -7° C may be used unless otherwise specified and is as follows:

8.2.2.1.1 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.

- 8.2.2.1.2 Samples received by the laboratory unfrozen and at 0 - 6° C, and are within 48 hours of collection may be frozen to less than -7° C at the time of receipt.
- 8.2.2.1.3 Allow samples to come to room temperature before analysis. All samples must be analyzed within 14 days of collection.
- 8.2.2.1.4 Thawed samples must be prepared and analyzed within 24 hours of thawing.
- 8.2.3 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.
- 8.2.4 The following steps apply for high concentration soil samples collected and preserved in the field with methanol as described in Section 8.2.
- 8.2.3.1 Use pre-tared 40, 60 or 125 mL jars with screw cap lids and PTFE 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 ampoule of purge and trap methanol with each sample container.
- 8.2.5 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.
- 8.3 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 wash 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 0 - 6° 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, 10 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 10 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 0 - 6° 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 0 - 6° 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 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™, ISCO 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 AND TROUBLESHOOTING

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 (CH₃OH), 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 (NaHSO₄), certified

11.3.2 Weigh 100 g of NaHSO₄ into a 500 mL volumetric flask. Mix to dissolve the NaHSO₄ 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) NaHSO₄.

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% NaHSO₄ 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. 2-chloroethylvinyl ether is not reported when sodium bisulfate is 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 0 - 6° 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 0 - 6° 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 for 20 minutes.
- 13.3.6 After shaking and sonicating, place in a refrigerator at 0 - 6° 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 0 - 6° C until analysis.

13.4 Prepare oily waste samples soluble in a water-miscible solvent as follows:

- 13.4.1 Place a 2 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 0 - 6° 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 $\frac{3}{4}$ 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 restrictions 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

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**Attachment 23.1
 Volatile Soil Sample Receipt Information Spreadsheet, Page 1**



Volatile Soil Sample Receipt Information Sheet

Client: _____ Date Received: 08/29/2003 Sheet Completed By: _____
 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?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Were Samples Received Non-Preserved in Encore Samplers?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
If Received in Encore Samplers, Was Sample Received Within 48 Hours?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Volatile Lab Informed That Samples Must Be Preserved AND Analyzed Within 48 Hours?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

High Level Soils

	Yes	No	N/A
Extra Sample Containers Received for Dry Weight Determination?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
MeOH Trip Blank Received (if MeOH not added from sealed ampules)?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Samples Collected in Which of the Following Ways?			
<u>Tared 40, 60, or 120 mL Containers:</u>			
Were Containers Supplied by TriMatrix?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Were Samples Received Within 4 Days After Collection, and Were They MeOH Preserved?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<u>Packed With No Headspace into Brass Tubes:</u>			
Were Samples Received Within 4 Days After Collection and Preserved Within 2 Hours of Sample Receipt?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<u>Packed With No Headspace in En Core Samplers:</u>			
Were Samples Received Within 40 Hours After Collection and Preserved Within 48 Hours of Collection?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Follow-Up

	Yes	No	N/A
Has Soil Weight Table Been Completed?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Has Project Chemist Been Informed of any Out of Compliance Samples?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

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**Attachment 23.2
 Volatile Soil Sample Receipt Information Spreadsheet, Page 2**



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	43.0			10.1			Accept Sample		
7	341548	40	25.3	10	43.0			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		

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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 0 - 6° C until transport to the laboratory.
- 2.2 When received by the laboratory, samples remain stored at 0 - 6° 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 $+ \pm 2$ 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 unsuitability for low level analyses, 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 0 - 6° 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 0 - 6° C until analysis.

Note: The methanol must be withdrawn from the sample within 24 hours of extracting the sample to keep 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:

8.3.1 Pipet 1.0 mL of extract into a 50 mL volumetric flask approximately $\frac{3}{4}$ full of laboratory reagent water (organic-free).

8.3.2 Dilute to volume with laboratory reagent water (organic-free).

8.3.3 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 within the calibration range.

8.5 Follow the main body of the procedure to complete analysis and data reduction.



STANDARD OPERATING PROCEDURE

Organic Carbon Walkley-Black

Agronomy, Methods of Soil Analysis 29-3.5.2

APPROVALS:

Area Supervisor: Heather L. Brady Date: 9-23-11
Heather L. Brady

QA Officer: Tom C. Boocher Date: 9-22-11
Tom C. Boocher

President: Douglas E. Kriscunas Date: 9-26-11
Douglas E. Kriscunas

Procedure Number: GR-06-105
Revision Number: 2.5

Date Initiated: 7/29/97
Effective Date: 9/30/11

Date Revised: 9/22/11
Pages Revised: All

By: Heather L. Brady

Total Number of Pages: 14

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 approximating organic carbon in non-carbonized soils and sediments, with a zero average organic carbon oxidation state.
- 1.2 The minimum reporting limit is 0.1% (dry weight) as organic carbon according to the following method requirements:

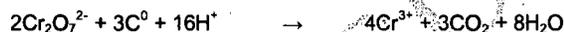
$$\text{Minimum Reporting Limit, \% C}^0 = \frac{10 \text{ mg C}^0}{10\text{g Soil}} \times 100\%$$

2.0 PRINCIPLE METHOD REFERENCES

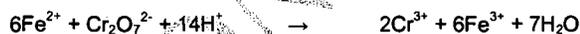
- 2.1 *Methods of Soil Analysis Part 2, Chemical and Microbiological Properties, 2nd Edition, Number 9 (Part 2), Agronomy, 1982, Section 29-3.5.2, "Walkley-Black Procedure"*

3.0 SUMMARY OF PROCEDURE

- 3.1 Organic carbon is determined by an oxidation-reduction reaction in which potassium dichromate is added to a sample, followed by addition of concentrated sulfuric acid. Dichromate ($\text{Cr}_2\text{O}_7^{2-}$) oxidizes organic carbon to CO_2 in an acidic medium as follows:



- 3.2 The reduced dichromate is quantitatively related to oxidized organic carbon. Any remaining $\text{Cr}_2\text{O}_7^{2-}$ is reduced by Fe^{2+} from the ferrous sulfate titrant. The endpoint at which all $\text{Cr}_2\text{O}_7^{2-}$ has been reduced is indicated by the maroon color of an o-phenanthroline ferroin indicator.



4.0 PARAMETER OR COMPOUND LIST

- 4.1 Organic Carbon

5.0 REFERENCED SOPs

- 5.1 TriMatrix SOP GR-15-109, *Laboratory Waste Disposal*, latest revision
- 5.2 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

- 6.1 Significant chloride (Cl^-) concentration in soil will give a positive error.

- 6.2 Fe^{2+} in soil will also give a positive error. Oven drying soils at 103 to 105° C oxidizes Fe^{2+} to Fe^{3+} and a more accurate organic carbon result can be determined. To minimize interference from Fe^0 , samples must not be ground or sieved with ferrous equipment.
- 6.3 Manganese oxide (MnO_2) may give a negative error. However, only a small percentage of the total MnO_2 actually takes part in the redox reaction. This reaction error should not be serious enough to need correcting for most matrices.
- 6.4 Analysis of carbonized materials such as charcoal, graphite, coal, and soot containing large quantities of elemental carbon is beyond the scope of this procedure. Soil analysis must only be performed on soil or sediment presumed to have an average carbon oxidation state of zero.

7.0 SAFETY PRECAUTIONS

- 7.1 Wear a laboratory coat and approved safety glasses while in the 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.
- 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 exposure 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 Waste samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-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 Soils must be collected in inert bottles (plastic or glass), with no preservative added.
- 8.2 Analysis must be performed within 28 days of sample collection.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

- 9.1 Burette, 50 mL, type A
- 9.2 Erlenmeyer flasks, 500 mL
- 9.3 Type A volumetric pipet, 10 mL
- 9.4 Sieve (non-ferrous), 0.5 mm
- 9.5 Mortar & pestle, porcelain or other non-ferrous material
- 9.6 Volumetric flasks, Type A
- 9.7 Macropipette
- 9.8 Bottle top dispenser for concentrated H₂SO₄, capable of dispensing 20 mL
- 9.9 Stir bars
- 9.10 Stir plate, unheated
- 9.11 Balance, top loading, capable of weighing to at least 0.01 g

- 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, ASTM Type II, Milli-Q system
- 11.2 Potassium dichromate (K₂Cr₂O₇), ACS, 1N
 - 11.2.1 In a 1,000 mL volumetric flask, dissolve 49.04 g of ACS grade K₂Cr₂O₇ (dried at 103 - 105° C)
 - 11.2.2 Dissolve the dichromate by mixing or swirling, then dilute to volume with laboratory reagent water.
 - 11.2.3 Expiration is six months from the date made.
- 11.3 Sulfuric acid (H₂SO₄), concentrated (18M), ACS
- 11.4 Ferroin indicator (0.025 M Phenanthroline Ferrous Sulfate Complex), purchased commercially
- 11.5 Ferrous sulfate heptahydrate (FeSO₄·7H₂O) ACS grade solution (0.5N)

-
- 11.5.1 In a 1000 mL volumetric flask, dissolve 140 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in laboratory reagent water.
- 11.5.2 Add 15 mL of concentrated H_2SO_4 .
- 11.5.3 Cool and dilute to volume with laboratory reagent water.
- 11.5.4 Expiration is three months from the date made unless the normality check of Section 14.2 is unacceptable.
- 11.6 Potassium hydrogen phthalate or KHP ($\text{HOOC-C}_6\text{H}_4\text{-COOK}$), acidimetric standard ACS, oven-dried
- 11.6.1 Place approximately 25 g KHP in a glass beaker and dry at 103-105° C for 2 hours.
- 11.6.2 Remove from the oven and immediately place in a desiccator. Cool to room temperature in the desiccator then transfer to a permanent storage bottle.
- 11.6.3 Dried KHP will not need re-dried if stored in the desiccator and is only opened to remove material.
- 11.6.4 Dispose of and replace dried KHP annually.
- 11.7 Ottawa sand – dried @ 550°c for 1 hour.
- 12.0 STANDARDS PREPARATION**
- 12.1 Laboratory control sample (10,000 mg/L C^0): Dissolve 2.128 g oven-dried KHP in a 100 mL volumetric flask with laboratory reagent water. Dilute to volume with reagent water and store at 0 - 6° C in an amber bottle. Expiration is six months from the date made.
- 12.2 Record all standard preparations in the LIMS system (Element™), each with a unique ID number. Refer to Attachment 23.1 for an example of analytical standards record.
- 13.0 SAMPLE PREPARATION**
- 13.1 Samples must be dried at 103 - 105° C until sufficiently dry to be ground into powder and sieved. Typically, the drying time is overnight. Record drying time data on the raw data spreadsheet (refer to Attachment 23.2), including the following:
- 13.1.1 Balance used
- 13.1.2 Date and time oven drying begins
- 13.1.3 Date and time oven-drying ends
- 13.1.4 Dish mass, in g
-

- 13.1.5 Pre-dried soil and dish mass, in g
- 13.1.6 Post-dried soil and dish mass, in g
- 13.2 Grind the dried soil in a porcelain mortar and pestle.
- 13.3 After grinding, pass through a 0.5 mm non-ferrous sieve.
- 13.4 Subsample the sieved material for analysis.

14.0 CALIBRATION PROCEDURES

- 14.1 Perform a titration blank (without soil or Ottawa sand) before processing samples to standardize the ferrous sulfate normality as follows:
 - 14.1.1 Add 10.0 mL of 1N $K_2Cr_2O_7$ to a 500 mL Erlenmeyer flask.
 - 14.1.2 Carefully add 20 mL of concentrated H_2SO_4 , directing the stream into the solution. Immediately, swirl gently until the reagents mix. This must be done inside a fume hood.
 - 14.1.3 Let the flask sit for approximately 30 minutes then add 200 mL of laboratory reagent water.
 - 14.1.4 Add 5-10 drops of ferroin indicator and titrate with 0.5N $FeSO_4$. The solution will turn bluish-green when approaching the endpoint. When the solution turns dark green, add titrant dropwise until the color changes sharply to maroon, which is the endpoint.
 - 14.1.5 Read and record the titrant volume used to at least the nearest 0.05 mL.
- 14.2 If titrant normality is not $0.500 \pm 0.05N$, clean the burette and remake the titrant solution and/or the 1N $K_2Cr_2O_7$.
- 14.3 Calculate ferrous sulfate normality based on the blank (refer to section 16.2).

15.0 ANALYTICAL PROCEDURE

- 15.1 Transfer up to 10.00 g of sieved material containing 10 to 25 mg organic carbon to a 500 mL Erlenmeyer flask.
- 15.2 Pipette 10.0 mL of 1N $K_2Cr_2O_7$ into the flask and gently swirl to disperse the soil into a suspension.
- 15.3 Carefully add 20 mL of concentrated H_2SO_4 , directing the stream into the suspension. Immediately, swirl gently until the soil and reagents mix. Place the flask on a magnetic plate, add a stir bar, and stir for one minute. This must be done inside a fume hood.

- 15.4 Let the flask sit for approximately 30 minutes.
- 15.5 After 30 minutes add 200 mL of laboratory reagent water. At this point filter the suspension if the endpoint will not be clearly discernable.
- 15.6 Add 5-10 drops of ferroin indicator and titrate with 0.5N FeSO₄. The solution will turn bluish-green when approaching the endpoint. When the solution turns dark green, add titrant dropwise until the color changes sharply to maroon.
- 15.7 Repeat a titration with less sample if less than 4.0 mL of titrant is used to reach the endpoint.
- 15.8 All results must be recorded on the raw data spreadsheet (Refer to Attachment 23.2). After titration is complete, empty the flask contents into the waste bottle labeled "TOC soil waste", to be disposed of as dichromate waste. Make sure waste bottles are labeled as dichromate waste, with the date filled on the label as well.

16.0 CALCULATIONS AND DATA HANDLING

- 16.1 All measurements and reagents used must be recorded on the raw data spreadsheet (refer to Attachment 23.2). Data must then be entered into a spreadsheet (refer to Attachment 23.3) to calculate the final concentration. The spreadsheet duplicates the logbook format for ease of data entry.
- 16.2 Normality of the FeSO₄ titrant is determined from the standardization as follows:

$$\text{normality of FeSO}_4 \text{ titrant} = \frac{10}{\text{mL of titrant}}$$

- 16.3 Percent organic carbon is calculated by the spreadsheet, as follows:

$$\text{Organic C, \%} = \frac{[10 - (\text{Normality of FeSO}_4 \times \text{mL of FeSO}_4)](0.003)(100)}{\text{g dried soil}} \times 1.3$$

Note: The BS should not include the 1.3 correction factor

17.0 DATA REPORTING AND DELIVERABLES

- 17.1 The following must be recorded on the preparation batch report or elsewhere for each batch analyzed:
 - 17.1.1 The BS, BLK, DUP and sample final concentration with units.
 - 17.1.2 Work order, date prepared, analyst, initials of the peer reviewer, stock standard numbers.
 - 17.1.3 Any batch comments that need made.

17.2 Refer to Attachment 23.4 for a preparation batch report example.

18.0 QUALITY ASSURANCE

18.1 Method quality control (QC) must be analyzed with each analysis batch of up to 20 samples. Matrix QC must be analyzed every 10 samples or with each analysis batch of less than 10 samples, whichever is more frequent.

18.1.1 Method QC consists of the Blank Spike (BS) and a method blank (BLK).

18.1.1.1 The approved BS is 1.0 mL of 10,000 mg/L C⁰ (refer to Section 12.1) pipetted into a 500 mL Erlenmeyer flask containing 1.0 g of dried Ottawa sand and analyzed as a sample. The expected value is 1.0 %. Acceptance limits are the control limits listed in Element. The expected value is calculated as follows:

$$\% C^0 = \frac{2.128 \text{ g KHP}}{100 \text{ mL}} \times \frac{\text{mole KHP}}{204.23 \text{ g KHP}} \times \frac{8 \text{ mole } C^0}{\text{mole KHP}} \times \frac{12.011 \text{ g } C^0}{\text{mole } C^0} \times \frac{1.0 \text{ mL}}{1.0 \text{ g Sand}} \times 100\%$$

18.1.1.2 A BLK is 10 g dried Ottawa sand analyzed as a sample. Blank results must be less than the reporting limit. If a blank is not less than the reporting limit, address the contamination problem then perform an acceptable blank.

18.1.2 Matrix QC consists of a digestion/titration duplicate (DUP).

18.1.2.1 A DUP is a second aliquot of dried sample analyzed the same as the first.

18.1.2.2 Acceptance limits for DUP percent difference are the control limits listed in Element™.

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) is required annually.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Prepare and analyze four blank spikes as samples (Section 18.1.1.1). Process the four spikes following every step in the procedure. Calculate average percent recovery and relative standard deviation of the average. Average percent recovery must be between within the BS acceptability window listed in Element™. Relative standard deviation must be less than 20%.

19.1.1.2 If either criterion is not met, locate and correct the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure and/or techniques used. If this occurs, correct the procedure or techniques and repeat the study successfully.

19.1.1.3 Samples may not be analyzed by any analyst until an initial demonstration of capability study has been successfully completed. Copies of successful IDC spreadsheets must be submitted to the Quality Assurance Department for training documentation.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A CDC study must be performed annually by all analysts using this procedure.

19.1.2.2 The CDC study may be completed by repeating the IDC, by processing four consecutive BS results obtained from routine sample analysis, by using the last four results from the annual MDL study or by successfully analyzing a performance testing study result.

19.1.2.3 Input CDC data to the same demonstration of capability spreadsheet and submit to the Quality Assurance Department for training documentation.

19.2 A Method Detection Limit (MDL) Study is required 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 outlined in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Methods of Soil Analysis Part 2, Chemical and Microbiological Properties, 2nd Edition, Number 9 (Part 2), Agronomy, 1982, Section 29-3.5.2, "Walkley-Black Procedure"*

23.0 ATTACHMENTS

23.1 Analytical Standards Record Example

23.2 Raw Data Example

23.3 Calculation Spreadsheet Example

23.4 Preparation Batch Report Example

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SOP Name: Soil Organic Carbon, Walkley – Black
Agronomy, Methods of Soil Analysis 29-3.5.2
SOP Number: GR-06-105

Revision Number: 2.5
Date Revised: 9/22/11
Date Initiated: 7/29/97

Attachment 23.1
Analytical Standards Record Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
1060787

Description:	TOC 10,000PPM	Expires:	Dec-18-11
Standard Type:	Analyte Spike	Prepared:	Jun-18-11
Solvent:	Solvent Lot #	Prepared By:	Heather L. Brady
Final Volume (mls):	100	Department:	Inorganic - Wet Chemistry
Vials:	1	Last Edit:	Jul-07-11 14:53 by HLB

2.128g KHP(9040497) into 100mls

Analyte	CAS Number	Concentration	Units
UV-254		146	ppm
Total Organic Carbon		10000	ppm
Organic Carbon	7440-44-0	10000	ppm
Carbon, Total Organic	7440-44-0	10000	ppm



SOP Name: Soil Organic Carbon, Walkley - Black
 Agronomy, Methods of Soil Analysis 29-3.5.2
 SOP Number: GR-06-105

Revision Number: 2.5
 Date Revised: 9/22/11
 Date Initiated: 7/29/97

Attachment 23.4
Preparation Batch Report Example

Trimatrix Laboratories, Inc.

PREPARATION BATCH 0606877 Page 1 of 1
 Inorganic - Wet Chemistry, Soil, Method-Specific Preparation
 (No Surrogate)
 Batch Comments: (none)

LV4
 Du 6-19-06
 6-19-06
 Printed: 6/17/2006 2:27:45PM

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0606082	TOC MSA 29-3.5.2				

Lab Number	Container	Prepared	By	Initial (g)	Final (mL)	nL Surrogate	Source ID	Spike ID	nL Spike	Client / QC Type	Extraction Comments
0606877-BLK1		Jun-16-06 08:00	HLB	10	10					BLANK	
0606877-DUP1		Jun-16-06 08:00	HLB	0.98	0.98		0606082-02			DUPLICATE	
0606877-BS1		Jun-16-06 08:00	HLB	0.5	0.5			A602089	500	LCS	
0606877-MS1		Jun-16-06 08:00	HLB	0.97	0.97		0606082-02	A602089	500	MATRIX SPIKE	
0606082-01	A	Jun-16-06 08:00	HLB	1.01	1.01						
0606082-02	A	Jun-16-06 08:00	HLB	0.97	0.97						
0606082-03	A	Jun-16-06 08:00	HLB	0.98	0.98						
0606082-04	A	Jun-16-06 08:00	HLB	1.13	1.13						
0606082-05	A	Jun-16-06 08:00	HLB	0.3	0.3						
0606082-06	A	Jun-16-06 08:00	HLB	0.88	0.88						
0606082-07	A	Jun-16-06 08:00	HLB	0.91	0.91						
0606082-08	A	Jun-16-06 08:00	HLB	1.14	1.14						
0606082-09	A	Jun-16-06 08:00	HLB	0.34	0.34						
0606082-10	A	Jun-16-06 08:00	HLB	0.87	0.87						
0606082-11	A	Jun-16-06 08:00	HLB	0.47	0.47						
0606082-12	A	Jun-16-06 08:00	HLB	0.38	0.38						
0606082-13	A	Jun-16-06 08:00	HLB	1.04	1.04						
0606082-14	A	Jun-16-06 08:00	HLB	0.85	0.85						
0606082-15	A	Jun-16-06 08:00	HLB	0.74	0.74						
0606082-16	A	Jun-16-06 08:00	HLB	0.85	0.85						
0606082-17	A	Jun-16-06 08:00	HLB	0.74	0.74						
0606082-18	A	Jun-16-06 08:00	HLB	0.88	0.88						
0606082-19	A	Jun-16-06 08:00	HLB	0.89	0.89						
0606082-20	A	Jun-16-06 08:00	HLB	1.1	1.1						

Comments:	Analyst Initials:
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STANDARD OPERATING PROCEDURE

Potentiometric pH

SW-846 9040C Standard Methods 4500-H⁺ B

APPROVALS:

Area Supervisor: Heather L. Brady Date: 1-12-12
Heather L. Brady

QA Officer: Tom C. Boocher Date: 1-12-12
Tom C. Boocher

Laboratory President: Douglas E. Kriscunas Date: 1-12-2012
Douglas E. Kriscunas

Procedure Number: GR-07-100
Revision Number: 3.5

Date Initiated: 10/5/05
Effective Date: 1/25/12

Date Revised: 1/12/12
Pages Revised: All

By: Tom C. Boocher

Total Number of Pages: 15

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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 drinking water, ground water, wastewater and wastes with greater than a 20% water phase by volume.
- 1.2 The effective measurement range is 2.0 to 12.5 pH units.
- 1.3 The minimum reporting limit is 2.0 pH units.

Note: Results of pH less than 2.0 or greater than 12.5 might indicate the sample source to be hazardous for corrosivity and/or not a permitted effluent. Consequently, the laboratory only reports such samples as "< 2.0" or "> 12.5" based on the sample and analysis calibration limits of the pH instrument.

2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IIIB, Revision 3, November, 2004, Method 9040C, "pH Electrometric Measurement"*
- 2.2 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, 4500-H⁺ B, "pH Value, Electrometric Method"*

3.0 SUMMARY OF PROCEDURE

- 3.1 Sample pH is measured potentiometrically using a buffer-calibrated combination reference/pH electrode and pH meter.

4.0 PARAMETER OR COMPOUND LIST

- 4.1 Potentiometric pH

5.0 REFERENCED SOPs

- 5.1 TriMatrix SOP GR-05-102, *Waste Disposal*, latest revision
- 5.2 TriMatrix SOP GR-07-113, *pH Potentiometric Method Soil and Waste (Non-Aqueous Liquids)*, latest revision
- 5.3 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision
- 5.4 TriMatrix SOP GR-10-114, *Thermometer Calibration and Verification*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

- 6.1 High sodium samples with very low or high pH will give incorrect meter readings. For pH results greater than 10.0, pH will be biased low unless using a "low sodium error" electrode. For pH less than 2.0, measurements will be biased high and must be reported as "pH <2.0". The electrode used has very little sodium error at low-sodium concentrations and this is not a factor with most environmental samples analyzed. However, report pH results greater than 12.5 as "pH >12.5".
- 6.2 Temperature variation between calibration buffers and samples is a source of measurement error. Buffers and samples must be measured at the same temperature.
- Note: The temperature sensor of the meter must be verified as accurate, at least annually, in accordance with TriMatrix SOP GR-10-114.
- 6.3 Soaps, oily matter, suspended solids or precipitates may coat the glass pH electrode and cause a sluggish response. Clean the electrode as follows:
- 6.3.1 For general cleaning, soak the pH electrode in 0.1M hydrochloric acid (HCl) or 0.1 nitric acid (HNO₃) for 15 minutes. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.
- 6.3.2 For grease and oil deposits, rinse with a mild detergent or methanol solution. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.
- 6.3.3 For inorganic deposits, rinse with 0.1M tetrasodium EDTA solution. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.
- 6.3.4 For protein deposits, digest with 1% pepsin in 0.1M HCl. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.
- 7.0 SAFETY PRECAUTIONS**
- 7.1 Wear a laboratory coat and approved safety glasses while in the 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.
- 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 exposure 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 Environmental samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-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 Collect samples in a plastic or glass bottle large enough to hold at least 100 mL. Fill the container to capacity when bringing to the laboratory for analysis.

8.2 Do not add preservative.

8.3 Analyze as soon as possible after receipt by the laboratory.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Potentiometric pH meter.

9.2 Sample cups, disposable, 50 ml

9.3 Magnetic stir plate, not heated

9.4 Stir bars, PTFE-coated

9.5 pH electrode, glass, with sodium error of ± 0.01 pH units at pH 12.0 in 0.5N sodium chloride solution

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.2 The pH electrode must be inspected frequently and refilled with fluid if necessary.

10.1 When necessary, clean the pH electrode in accordance with Section 6.3.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, ASTM Type II, Milli-Q system

11.2 Calibration buffers, commercially purchased

11.3 Quality control buffers, commercially purchased

11.4 Hydrochloric acid, reagent grade or better

12.0 STANDARDS PREPARATION

12.1 There is no standards preparation directly associated with this procedure.

13.0 SAMPLE PREPARATION

13.1 Samples must be brought to ambient temperature before analysis. If sample temperature differs from calibration buffers by more than $\pm 2^{\circ}\text{C}$, a temperature correction must be made (manually or automatically).

13.2 Further sample preparation is not required if samples have a greater than 20% aqueous phase. Analysis of pH samples with less than 20% (v/v) aqueous phase is beyond the scope of this procedure. Such samples need re-logged in for analysis by TriMatrix SOP GR-07-113 by the project chemist.

13.3 The aqueous phase in a sample can be determined by visually observing the sample jar and estimating whether approximately 20% (v/v) is a water phase by volume. If there is obviously at least 20% (v/v) free water in the sample then determine pH on the water phase only. If there is obviously less than 20% (v/v) water or no water then proceed in accordance with Section 13.2. For less ambiguity, determine the water phase volume before shaking the sample.

14.0 CALIBRATION PROCEDURES

14.1 Calibrate the pH meter to bracket expected pH values with three buffers, three pH units or more apart. Refer to Attachment 33.5 for a pH calibration flowchart.

Note: When field sample pH is between 10.0 and 12.5, control the temperature to $25 \pm 1^{\circ}\text{C}$.

14.2 Use only fresh buffer solution for the calibration. Do not save to use on another meter. Discard immediately after the calibration is complete.

14.3 To calibrate the pH meter for normal-range samples, use buffers 4.0, 7.0 and 10.0.

14.3.1 If a sample reads less than pH 4.0, recalibrate using buffers 2.0, 4.0, and 7.0 then reanalyze.

14.3.2 If a sample reads greater than pH 10.0, recalibrate using buffers 7.0, 10.0 and 12.0 then reanalyze.

Note: Always begin the calibration with the pH 7.00 buffer solution.

14.4 When calibrating, wait for a stable reading for each buffer solution.

14.5 During calibration and analysis, gently stir each solution at a constant rate great enough to minimize drift (<0.1 pH unit) but not enough to generate bubbles and/or aeration that might introduce carbon dioxide.

14.6 Record the displayed calibration slope on the benchsheet and in the run logbook.

Note: New Accumet electrodes—fresh out of the box—have a slope between 95.0% and 102%. If the slope drops below 92%, clean the electrode. If the slope remains below 90.0% or above 102% after cleaning, replace the electrode.

14.7 Once the meter is calibrated, read each buffer as the initial calibration verification (CCV). Each must read correct to within ± 0.05 pH units. If not, repeat the calibration until all buffers read within ± 0.05 pH units.

14.8 After calibrating and verifying the calibration, the meter is ready for sample analysis.

15.0 ANALYTICAL PROCEDURE

15.1 For all analyses, adjust the probe clamp so immersion will be just deep enough into the solution to establish good electrical contact through the electrode fiber-capillary hole. Rinse probes thoroughly with reagent water before transferring to another solution. Discard the rinseate.

Note: Standard and sample temperatures must be within $\pm 2^\circ$ C of the calibration buffer temperature. Also, do not let samples sit open on the bench for any length of time or over-stir when taking a reading as exposure to the air and/or excessive stirring will introduce carbon dioxide and lower the pH.

15.2 If not already done, verify the calibration by checking each calibration buffer. Record these checks as CCV₁, CCV₂ and CCV₃. Gently stir at the same rate used during calibration. Wait for the meter to beep and display "stable" then record the pH to the second decimal place. Each value obtained must be within ± 0.05 pH units of the certified value for acceptability. If acceptable, the meter is ready for sample analysis. Remember to rinse the probe thoroughly with reagent water between solutions.

15.3 Repeat with second-source verification (SRM₁, SRM₂) buffers as specified in Attachment 23.5. These readings must also be within ± 0.05 pH units of the certified value for acceptability. Sample analysis may not begin until CCV and SRM results are acceptable.

15.4 After acceptable CCV and SRM results are obtained, measure the pH of up to 10 samples (this count includes the SRM). If necessary, repeat sample analysis on multiple volumes of sample until the difference in readings is less than 0.1 pH units. Two or three volumes are usually sufficient. Report the value obtained.

15.5 After 10 analyses and at the end of each batch, analyze a continuing calibration verification (CCV) as specified in Attachment 23.5. The CCV will be the same three buffers used to calibrate the instrument. Measured results must be within ± 0.05 pH units of the certified value for acceptability.

15.6 Provided the three CCV buffers pass, sample analysis may continue with the three CCV buffers being analyzed after every 10 analyses. Also, close the analytical batch with an acceptable CCV check of the three buffers.

15.7 If a CCV fails, the previous 10 samples must be reanalyzed after cleaning the electrode in accordance with Section 6.3, and recalibrating.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The pH meter measures all data against the calibration and displays a direct pH reading. No manual calculations are necessary.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts are responsible for data quality and for correctly filling in all documentation. This is required for quality control and to provide clients with fully defensible data.

17.2 Record the following data and all other data specified throughout the procedure on the benchsheet and/or in the logbook for each batch handled in:

17.2.1 The laboratory information management system (LIMS/Element™) standard identification of all calibration buffers used (logbook)

17.2.2 Sample temperature (benchsheet)

17.2.3 The pH result (benchsheet)

17.2.4 Observed values of each buffer used for calibration (benchsheet)

17.2.5 The slope (benchsheet)

17.2.6 Method name and number (benchsheet)

17.2.7 Refer to Attachment 23.1 for a benchsheet example.

17.3 Instrument logbooks must be filled in with the following information.

17.3.1 Date analyzed

17.3.2 Analyst's initials

17.3.3 Calibration standards used

17.3.4 Client name and sample numbers analyzed

Note: Refer to Attachment 23.3 for an instrument logbook example.

17.4 All logbooks must be filled in completely and correctly. All corrections must be made in indelible ink. **Corrections must be made with a single dated/initialed lineout. Write-overs are not acceptable.** An incorrectly entered result must remain legible even after the correction is made. A new result must be placed near the incorrect result with the date and analyst's initials written next to it. Blank lines in logbooks must be Z'd out.

17.5 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in correctly and completely.

18.0 QUALITY ASSURANCE

18.1 Method and matrix quality control must be analyzed with each batch of up to 10 samples.

18.1.1 Quality control consists of two secondary calibration verification (SRM) buffers, an initial calibration verification (as the three CCV buffers) and continuing calibration verifications (the same three CCV buffers) as specified in Attachment 23.5. Acceptance limits are ± 0.05 pH units of the certified value.

18.1.1.1 Acceptance limits are ± 0.05 pH units of the certified value.

18.1.1.2 Include quality control samples in the frequency count.

pH	Method \pm limit	Lower Control Limit as pH (LCL)	Upper Control Limit as pH (UCL)	LIMS ECL (%)	LIMS UCL (%)
2	0.05	1.95	2.05	97.5	102.5
4	0.05	3.95	4.05	98.8	101.3
6	0.05	5.95	6.05	99.2	100.8
8	0.05	6.95	7.05	99.3	100.7
10	0.05	7.95	8.05	99.4	100.6
12	0.05	9.95	10.05	99.5	100.5
14	0.05	11.95	12.05	99.6	100.4

18.1.2 Matrix quality control consists of a sample duplicate (DUP) every 10 samples. A DUP is an analysis replicate analyzed in the same way as the original. Acceptance limits in relative percent difference (RPD) are listed in the laboratory information management system (Element™).

18.2 Refer to TriMatrix GR-10-106 for the appropriate corrective action needed when an out-of-control condition exists.

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 study (IDC). While IDC studies are not instrument dependent, one is required on each pH meter used 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 Prepare four aliquots of pH 6.00 or 8.00 buffer. Process the four aliquots following every step in this procedure including calibration. Calculate average percent recovery and standard deviation of the average using the IDC spreadsheet located on the laboratory intranet library. The average value must be within ± 0.1 pH units of the certified value. The standard deviation must be $\leq 3\%$.

19.1.1.2 If these criteria are not met, locate and correct the source of the problem, and repeat the study. Repeated failure will confirm a general problem with the procedure or techniques used. If this occurs, locate and correct the procedure or inappropriate technique, then repeat the study successfully.

19.1.1.3 Samples may not be analyzed by any analyst on any pH meter until a demonstration of capability study has been successfully completed. Give a copy of successful studies, including the IDC spreadsheet and raw data to the quality assurance department for training documentation and review.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A demonstration of capability study must be performed annually by all analysts. This may be accomplished as follows:

19.1.2.1.1 By repeating the initial demonstration of capability study.

19.1.2.1.2 By processing four consecutive pH 6.00 or 8.00 results (4 of the same pH) exclusively obtained by the analyst during routine sample analysis.

19.1.2.1.3 By exclusively obtaining an acceptable blind performance evaluation study result obtained during routine sample analysis.

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 maintained on the laboratory intranet library.

21.2 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 IIIB, Revision 3, November, 2004, Method 9040C, pH Electrometric Measurement*

22.2 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, 4500-H⁺ B, "pH Value, Electrometric Method"*

23.0 ATTACHMENTS

23.1 Sample Benchsheet Example

23.2 Data Review Report Example

23.3 Instrument Logbook Example

23.4 Standards Log Example

23.5 pH Calibration Flowchart

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SOP Name: Potentiometric pH
 SW-846 Method 9040C, Standard Methods 4500-H⁺ B
 SOP Number: GR-07-100

Revision Number: 3.5
 Date Revised: 1/12/12
 Date Initiated: 10/5/95

Attachment 23.1
 Sample Benchsheet Example

TriMatrix Laboratories, Inc.

ANALYSIS SEQUENCE 6071943 Page 1 of 1

due 7/31
 Printed: 7/19/2006 5:57:05PM

Inorganic - Wet Chemistry, Water, Jul-19-06
 Instrument = 164, Calibration = UNASSIGNED

Sequence Analyses:
 pH 4500-H B

Lab Number	Analysis	Contain	pH STD ID	Temp. TEMP ID	Client / QC Type	pH	Temp
6071943-ICV1	QC		4.99	22.4	INITIAL CAL CHECK		
0608184-SRM1	QC		6.02	22.6	REFERENCE		
0608184-SRM2	QC		7.41	22.4	REFERENCE		
6071943-CCV1	QC		7.02	22.5	CALIBRATION CHECK		
0807223-01	pH 4500-H B	A	9.34	22.3	Environmental Resource Associates	9.34	22.1
0608184-DUP1	QC 0607223-01		9.34	22.1	DUPLICATE	9.34	21.9
6071943-CCV2	QC		7.00	22.6	CALIBRATION CHECK		

ICV (6.99 ÷ 7.00) (100%) = 99.9%

SRM1 (6.02 ÷ 6.00) (100%) = 100.3%

SRM2 (7.41 ÷ 7.100) (100) = 100.1%

CCV1 (7.02 ÷ 7.00) (100) = 100.3%

CCV2 (7.00 ÷ 7.00) (100) = 100%

Comments:	Analyst Initials:
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Attachment 23.2
Data Review Report Example
Trimatrix Laboratories, Inc.
Data Review Report -- Wet Chem Lab

Sequence = 6071943 Page 1 of 1

On 1/28/2008 at 5:14

<u>SampleID</u>	<u>Analysis</u>	<u>RResult</u>	<u>Diln</u>	<u>FResult</u>	<u>FMRL Qualifier</u>	<u>Recovery</u>	<u>RPD</u>	<u>Analyzed</u>	<u>Analyst</u>	<u>Batch</u>
6071943-ICV1	pH 4500-H B	6.99	1	6.99		100		7/19/2006 2:56	MSM	6071943
0608164-SRM1	pH 4500-H B	6.02	1	6.02	1.0	100		7/19/2006 2:56	MSM	0608164
0608164-SRM2	pH 4500-H B	7.41	1	7.41	1.0	100		7/19/2006 2:56	MSM	0608164
6071943-CCV1	pH 4500-H B	7.02	1	7.02		100		7/19/2006 2:56	MSM	6071943
0607223-01	pH 4500-H B	9.34	1	9.3	1.0 WCB			7/19/2006 2:56	MSM	0608164
0608164-DUP1	pH 4500-H B	9.34	1	9.34	1.0		0	7/19/2006 2:56	MSM	0608164
6071943-CCV2	pH 4500-H B	7	1	7.00		100		7/19/2006 2:56	MSM	6071943

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Attachment 23.3
 Instrument Logbook Example


Date	Analyst	Buffer Solution pH Concentrations and Lot Numbers (fill in Lot Numbers only for Buffers Used)					Calibration Verification Level and Lot Number		CV Results	Acceptable CV (±0.05 TV)	Client and Sample Numbers
		2	4	7	10	12	TV	Lot Number			
6/17/05	Jm7	DM4.79.16	DM4.42.12	DM2.99.10			6.00	DM4.78.4	5.93	yes/no	
6/20/05	Jm7		DM4.42.12	DM2.99.10	DM11.72.13		6.00	DM4.78.4	6.00	yes/no	
6/23/05	Jm7	DM4.79.16	DM4.42.12	DM2.99.10			6.00	DM4.78.4	6.02	yes/no	
6/23/05	Jm7				DM4.42.12		6.00	DM3.78.4	5.96	yes/no	
6/23/05	ASG		DM4.42.12	DM2.99.10	DM4.42.13		6.00	DM3.78.4	6.02	yes/no	05 06 199-01
6/29/05	A069	DM4.79.16	DM4.42.12	DM2.99.10			6.00	DM3.78.4	6.00	yes/no	392438
6/30/05	A069		DM4.42.12	DM2.99.10	DM4.42.13		6.00	DM3.78.4	6.04	yes/no	
6/30/05	A069		DM4.42.12	DM2.99.10	DM4.42.13		6.00	DM3.78.4	6.07	yes/no	
6/30/05	A069	DM4.79.16	DM4.42.12	DM2.99.10			6.00	DM3.78.4	6.07	yes/no	
							7.40	DM3.78.4	7.38	yes/no	
										yes/no	
										yes/no	
										yes/no	
										yes/no	
										yes/no	
										yes/no	
										yes/no	
										yes/no	
										yes/no	

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SOP Name: Potentiometric pH
 SW-846 Method 9040C, Standard Methods 4500-H⁺ B
 SOP Number: **GR-07-100**

Revision Number: 3.5
 Date Revised: 1/12/12
 Date Initiated: 10/5/95

**Attachment 23.4
 Standards Log Example**

**Analytical Standard Record
 TriMatrix Laboratories, Inc.
 A512818**

Description:	pH buffer 7.00 IN16.50-13	Expires:	Dec-30-06
Standard Type:	Calibration Star	Prepared:	Jan-06-06
Solvent:	Solvent Lot #	Prepared By:	** Vendor *
Final Volume (mls):	20000	Department:	Expired
Vials:	1	Last Edit:	Aug-10-06 10:10 by MSM

Fisher catalog # SB107-20
 Lot:054717-24

Analyte	CAS Number	Concentration	Units
pH		7	pH Units
Color (Apparent)		7	pH Units
Carbon Dioxide	124-38-9	7	pH Units
Alkalinity, Phenolphthalein		7	pH Units
Alkalinity, Total		7	pH Units
Alkalinity, Hydroxide		7	pH Units
Alkalinity, Carbonate		7	pH Units
Alkalinity, Bicarbonate		7	pH Units
Acidity as CaCO ₃		7	pH Units

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Attachment 23.5
pH Calibration Flowchart

pH Calibration and Quality Control

Range											LCS	
High	7						10				12	8,12
Mid	4		7				10					6,8
Low	2	4		7								2,6
pH	2	3	4	5	6	7	8	9	10	11	12	pH

- Run three point curve, ALWAYS starting with 7. Slope of the curve must be between 92 - 102%.
 High: 7, 10, 12
 Mid: 4, 10
 Low: 7, 2, 4
- Re-run each point of the curve as a sample as CCVs. Response must be ± 0.05 pH units of true value.
- Run the LCSs appropriate to the curve. Response must be ± 0.05 pH units of true value. The LCSs are second source buffers.
 High: 8, 12
 Mid: 6, 8
 Low: 2, 6
- Every 10 samples and at end of batch run CCVs. Response must be ± 0.05 pH units of true value. The CCVs are the same buffers as the curve.
 High: 10, 12
 Mid: 4, 10
 Low: 2, 4

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STANDARD OPERATING PROCEDURE

Potentiometric pH in Soil and Waste

SW-846 Method 9045D

APPROVALS:

Area Supervisor: Heather L. Brady Date: 1-12-12
 Heather L Brady

QA Officer: Tom C. Roche Date: 1-12-12
 Tom C. Roche

Laboratory President: Douglas E. Kriscunas Date: 1-12-2012
 Douglas E. Kriscunas

Procedure Number: GR-07-113
 Revision Number: 0.4

Date Initiated: 10/15/99
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By: Heather L. Brady

Total Number of Pages: 15

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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 solids, sludges, waste and non-aqueous liquids with less than a 20% (v/v) water phase.
- 1.2 The effective measurement range is 2.0 to 12.0 pH units.
- 1.3 The minimum reporting limit is 2.0 pH units.

Note: Results of pH less than 2.0 or greater than 12.5 might indicate the sample source to be hazardous for corrosivity and/or not a permitted effluent. Consequently, the laboratory only reports such samples as "< 2.0" or "> 12.5" based on the sample and analysis calibration limits of the pH instrument.

2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IIIB, Method 9045D, Revision 4, November, 2004 "Soil and Waste pH"*

3.0 SUMMARY OF PROCEDURE

- 3.1 Samples are mixed with water and pH of the water phase is measured potentiometrically using a buffer-calibrated combination reference/pH electrode and pH meter.

4.0 PARAMETER OR COMPOUND LIST

- 4.1 Potentiometric pH

5.0 REFERENCE SOPs

- 5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision
- 5.2 TriMatrix SOP GR-07-100, *Potentiometric pH*, latest revision
- 5.3 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision
- 5.4 TriMatrix SOP GR-09-128, *Homogenization, Grinding and Drying of Solid Samples*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

- 6.1 High sodium samples with very low or high pH will give incorrect meter readings. For pH results greater than 10.0, pH will be biased low unless using a "low sodium error" electrode. For pH less than 2.0, measurements will be biased high and must be reported as "pH <2.0". The electrode

used has very little sodium error at low-sodium concentrations and this is not a factor with most of the environmental samples analyzed. However, pH results greater than 12.0 must be reported as "pH >12.0".

6.2 Temperature variation between calibration buffers and samples is a source of measurement error. Buffers and samples must be measured at the same temperature as specified in the procedure.

6.3 Soaps, oily matter, suspended solids, or precipitates may coat the glass pH electrode and cause a sluggish response. The electrode must be kept clean as follows:

6.3.1 For general cleaning, soak the pH electrode in 0.1M hydrochloric acid (HCl) or 0.1 nitric acid (HNO₃) for 15 minutes. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

6.3.2 For grease and oil deposits, rinse with a mild detergent or methanol solution. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

6.3.3 For inorganic deposits, rinse with 0.1M tetrasodium EDTA solution. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

6.3.4 For protein deposits, digest with 1% pepsin in 0.1M HCl. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

7.0 SAFETY PRECAUTIONS

7.1 Wear a laboratory coat and approved safety glasses while in the 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.

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 exposure 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 Environmental samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-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 must be collected in a plastic or glass bottle large enough to hold at least 100 mL.

8.2 No preservative is added or required.

8.3 Samples not analyzed for pH in the field must be analyzed as soon as possible after receipt by the laboratory.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Potentiometric pH meter

9.2 Sample cups, disposable, 50 mL

9.3 Magnetic stir plate, not heated

9.4 Magnetic stir bars, PTFE-coated

9.5 The pH electrode used is Thermo Orion, model 9156APWP, serial 250984-A01, with sodium error of 0.01 pH units at pH 12.0 in 0.5N sodium chloride solution

9.6 Balance, top-loading, capable of measurement to 0.1 g

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 The pH electrode must be inspected frequently and refilled with fluid if necessary.

10.2 When necessary, clean the pH electrode in accordance with Section 6.3.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, ASTM Type II, Milli-Q

11.2 Calibration buffers, commercially purchased

11.3 Quality control buffers, commercially purchased

11.4 Hydrochloric acid, reagent grade or better

12.0 STANDARDS PREPARATION

12.1 There is no standards preparation directly associated with this procedure.

13.0 SAMPLE PREPARATION

13.1 Samples must be brought to ambient temperature before analysis. If sample temperature differs from calibration buffers by more than $\pm 2^\circ\text{C}$, a temperature correction cannot be made by the instrument.

13.2 Further sample preparation is required if samples have less than 20% (v/v) of aqueous phase. Analysis of pH samples with greater than 20% (v/v) aqueous phase is beyond the scope of this procedure. Such samples need re-logged in for analysis by Trimatrix SOP GR-07-100, by the project chemist.

13.3 The aqueous phase in a sample can be determined by visually observing the sample jar and estimating whether approximately 20% (v/v) is a water phase by volume. If there is obviously at least 20% (v/v) free water in the sample then determine pH on the water phase only. If there is obviously less than 20% (v/v) water or no water then proceed in accordance with Section 15.0. For less ambiguity, determine the water phase volume before shaking up the sample.

13.4 Prepare samples with less than 20% (v/v) water phase as follows:

13.4.1 Weigh out 20 g of sample into a 50 mL sample cup then add 20 mL of laboratory reagent water. Cover and continuously stir for five minutes without generating bubbles or effervescence. If working with a water-absorbing matrix, use 20 g of sample and 40 mL of laboratory reagent water.

13.4.2 Let the suspension stand for one hour (15 minutes for waste samples) to let suspended particles settle out. If a waste is multiphasic, separate and measure only the aqueous phase. Electrodes must be cleaned (Section 6.3) if coated with nonaqueous material.

13.4.3 After the suspension has settled and the water phase has been isolated (if necessary), check the pH.

Note: All standard and sample temperatures must be within $\pm 2^\circ\text{C}$ of the calibration buffer temperature.

CALIBRATION PROCEDURES

14.1 The pH meter must be calibrated to bracket expected pH values with three buffers, three pH units or more apart. Refer to Attachment 23.5 for a pH calibration flowchart.

Note: When field sample pH is between 10.0 and 12.0, control sample temperature to $25 \pm 1^\circ\text{C}$.

- 14.2 To calibrate the pH meter for normal-range samples, use buffers 4.0, 7.0 and 10.0.
- 14.2.1 If a sample reads less than pH 4.0, recalibrate using buffers 2.0, 4.0, and 7.0 then reanalyze.
- 14.2.2 If a sample reads greater than pH 10.0, recalibrate using buffers 7.0, 10.0 and 12.0 then reanalyze.
- Note: Always begin the calibration with pH 7.00 buffer solution.
- 14.3 Start calibration by pressing the mode key until the pH mode indicator is displayed. Place the two probes into the first buffer.
- 14.4 Press 2nd cal. ("calibrate", time and date of the last calibration will be displayed). After a few seconds P1 will be displayed in the lower field, indicating the meter is ready for the first buffer.
- 14.5 Wait for a stable reading. Manually enter the value using the scroll keys then press "yes". Enter a value by pressing the "A" or "V" key. The first digit will flash. Continue pressing the scroll key until the desired value is displayed. Press "yes" to accept. Continue for each digit. When the correct buffer value is displayed, press "yes" to enter.
- 14.6 The P2 prompt is then displayed for the second buffer.
- 14.7 Rinse the probes and place in the second buffer. Wait for a stable reading and enter the correct buffer value, then press "yes". The P3 prompt for the third buffer will then be displayed.
- 14.8 Rinse the probes and place in the third buffer. Wait for a stable reading and the correct buffer value, then press "yes".
- 14.9 When the P4 prompt is displayed, press "measure". Record the displayed calibration slope on the benchsheet and in the run logbook. The slope needs to be 90 – 102%. If not, clean the electrode. Read each buffer as a sample. (Each must read correct to within ± 0.05 pH units). If not, repeat the calibration until all buffers read within ± 0.05 pH units. When the meter is calibrated, it is ready for sample analysis.
- 14.10 All buffers must be used fresh. Do not save to use on another meter. Discard immediately after the calibration is complete.
- 14.11 During calibration and analysis, all buffer solutions and samples must be gently stirred at a constant rate great enough to minimize drift (< 0.1 pH unit) but not enough to generate bubbles and/or aeration that might introduce carbon dioxide into solution from the air.

15.0 ANALYTICAL PROCEDURE

- 15.1 Before checking the pH, adjust the probe clamp, so immersion will be just deep enough into the solution to establish good electrical contact through the electrode fiber-capillary hole. Rinse probes thoroughly with laboratory reagent water before transferring to another solution. Discard the rinseate.
-

Note: Standard and sample temperatures must be within $\pm 2^{\circ}$ C of the calibration buffer temperature. Also, do not let samples sit open on the bench or over-stir when taking a reading as exposure to the air and/or excessive stirring will introduce carbon dioxide and lower the pH.

- 15.2 Place the probe in the Initial Calibration Verification (ICV) buffer. An ICV is the midpoint buffer of the calibration (midpoints are 4.00, 7.00 or 10.00). Gently stir at the same rate used during calibration. Wait for the meter to beep and display "ready" then record the pH result to the second decimal place. The value obtained must be within ± 0.05 pH units of the certified value for acceptability. Rinse the probe thoroughly with reagent water.
- 15.3 Repeat with Laboratory Control Samples (LCS) as specified in Attachment 23.5. These readings must also be within ± 0.05 pH units of the certified value for acceptability. Sample analysis may not begin until ICV and LCS results are acceptable.
- 15.4 After acceptable ICV and LCS results are obtained, measure the pH of up to 10 samples (this count includes the LCS). Repeat sample analysis at multiple volumes until the difference in readings is less than 0.1 pH units. Two or three volumes are usually sufficient. Report the value obtained.
- 15.5 After 10 analyses, analyze a Continuing Calibration Verifications (CCV) as specified in Attachment 23.5. The CCV will be the same buffers used to calibration the instrument. The measured result must be within ± 0.05 pH units of the certified value.
- 15.6 Provided the CCV buffers pass, sample analysis may continue with a CCV being analyzed after every 10 analyses. The analytical batch must also be closed with an acceptable CCV.
- 15.7 If a CCV fails, the previous 10 samples must be reanalyzed. Before reanalyzing, clean the electrode in accordance with Section 6.3 then recalibrate.

16.0 CALCULATIONS AND DATA HANDLING

- 16.1 The pH meter measures all data against the calibration and displays a direct pH reading. No manual calculations are necessary.
- 16.2 Rules for significant figure reporting:
- 16.2.1 Report values to 0.1 pH units for samples (example: report 7.6, not 7.59).
 - 16.2.2 Report values to 0.01 pH units for quality control (example: report 7.63, not 7.6).
 - 16.2.2 Report pH of less than 2.0 as "pH <2.0". Do not report pH values less than 2.0.
 - 16.2.3 Report pH of greater than 12.0 as "pH >12.0". Do not report pH values greater than 12.0.

17.0 DATA REPORTING AND DELIVERABLES

- 17.1 Analysts processing sample batches are responsible for data quality and for correctly filling in all documentation. This is required for quality control and to provide clients with fully defensible data.
- 17.2 The following data and all other data specified throughout this procedure must be recorded on the Element™ printout and/or in the logbook for each batch handed in:
- 17.2.1 The standards logbook number of calibration buffers used (logbook)
 - 17.2.2 Sample temperature (benchsheet)
 - 17.2.3 The pH result (benchsheet)
 - 17.2.4 Observed values of each buffer used for calibration (benchsheet)
 - 17.2.5 The slope (benchsheet)
 - 17.2.6 Method name and number (benchsheet)
 - 17.2.7 Refer to Attachment 23.1 for an analysis sequence report example.
- 17.3 Instrument logbooks must be filled in for each batch handed in with the following information.
- 17.3.1 Date analyzed
 - 17.3.2 Analyst's initials
 - 17.3.3 Calibration standards used
 - 17.3.4 Client name and sample numbers analyzed
- Note: Refer to Attachment 23.3 for an instrument logbook example.
- 17.4 All logbooks must be filled in completely and correctly. All corrections must be made in indelible ink. **Corrections must be made with a single dated/initialed lineout. Write-overs are not acceptable.** An incorrectly entered result must remain legible even after the correction is made. A new result must be placed near the incorrect result with the date and analyst's initials written next to it. Blank lines in logbooks must be Z'd out.
- 17.5 If internal chain-of-custody (COC) is required, it is very important that the COC form be filled in correctly and completely.

18.0 QUALITY ASSURANCE

- 18.1 Method and matrix quality control must be analyzed with each batch.
-

- 18.1.1 Quality control consists of two laboratory control samples (LCS), an initial calibration Verification (ICV) and continuing calibration verifications (CCV) as specified in Attachment 23.5. Acceptance limits are ± 0.05 pH units of the certified value.
- 18.1.1.1 Acceptance limits are ± 0.05 pH units of the certified value.
 - 18.1.1.2 Quality control samples need included in the frequency count.
- 18.1.2 Matrix quality control consists of a sample duplicate (DUP) run every 10 analyses. A DUP is an analysis replicate analyzed in the same way as the original. Acceptance limits in relative percent difference (RPD) are listed in the laboratory information management system (Element™).
- 18.2 Refer to TriMatrix GR-10-106 for the appropriate corrective action needed when an out-of-control condition exists.
- 19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION**
- 19.1 Before performing sample analysis, each analyst must first demonstrate the ability to generate acceptable results by running an initial demonstration of capability study. Also, while a demonstration of capability study is not instrument dependent, it is required for each pH meter as well. Analyst and pH meter capability studies must be repeated annually.
- 19.2 Prepare four aliquots of a soil sample following every step in the procedure. Have an experienced analyst independently prepare four aliquots of the same sample for comparison.
- 19.3 Calibrate the pH meter and analyze the four aliquots. Have the experienced analyst do the same with the comparison set.
- 19.4 Calculate average recovery (\bar{x}) and standard deviation of the average (s) in pH units by entering the eight results into the Smith-Satterthwaite spreadsheet for determining demonstration of capability acceptance. The spreadsheet is located on the laboratory intranet library.
- 19.4.1 The spreadsheet will calculate a statistical comparison between the two sets of data. If the spreadsheet determines the two analyst averages are statistically indistinguishable, the analyst and pH meter are authorized to run samples.
 - 19.4.2 If the spreadsheet indicates the two data sets are not indistinguishable, the analyst must proceed according to Section 19.5.
- 19.5 Locate and correct the source of the problem and repeat the test. Repeated failure however, will confirm a general problem with the procedure or technique used. If this occurs, locate and correct the procedure or technique, then repeat the test beginning with section 19.2. Samples may not be analyzed by any analyst or on any pH meter until a demonstration of capability study has been successfully completed. Copies of successful studies, spreadsheets and raw data must be given to the Quality Assurance department.

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19.6 A pH demonstration of capability study must be performed annually by all analysts. This may be accomplished by repeating the initial test or by running an acceptable performance testing study.

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. This information is located on the laboratory intranet library in the MSDS folder.
- 21.2 Follow all applicable instructions in GR-15-102 for Laboratory waste disposal.

22.0 REFERENCES

- 22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3^d Edition, Final Update IIIB, Method 9045D, Revision 4, November, 2004, "Soil and Waste pH"*

23.0 ATTACHMENTS

- 23.1 Analysis Sequence Report Example
- 23.2 Preparation Batch Report Example
- 23.3 Instrument Logbook Example
- 23.4 Standards Log Report Example
- 23.5 pH Calibration Flowchart



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Attachment 23.1
Analysis Sequence Report Example

TriMatrix Laboratories, Inc.

ANALYSIS SEQUENCE **6062935** Page 1 of 1

Printed: 5/30/2007 10:06:02PM

Inorganic - Wet Chemistry, Waste, Jun-29-06

Instrument = 164, Calibration = UNASSIGNED

Sequence Analyses:
pH 9045C

Lab Number	Analysis	Contain	STD ID	ISTD ID	Client / QC Type	Extraction Comments
6062935-ICV1	QC		A512818		INITIAL CAL CHECK	
0607475-SRM1	QC				REFERENCE	
0607475-SRM2	QC				REFERENCE	
6062935-CCV1	QC		A512818		CALIBRATION CHECK	
0606497-01	pH 9045C	A				
0607475-DUP1	QC				DUPLICATE	
6062935-CCV2	QC		A512818		CALIBRATION CHECK	

Comments:

Analyst
Initials:

seq_TriMatrix.rpt



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Attachment 23.2
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0703937 Page 1 of 1
Inorganic - Wet Chemistry, Waste, General Inorganic Prep
(No Surrogate)
Batch Comments: (none)

Printed: 5/30/2008 1:49:20PM

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0704160	pH 9045C				

Lab Number	Contain	Prepared	By	Initial (g)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0703937-DUP1		Apr-16-07 11:14	CLB	20	20		0704160-01			DUPLICATE	
0703937-SRM1		Apr-16-07 11:14	CLB	20	20			A608776	20000	REFERENCE	
0703937-SRM2		Apr-16-07 11:14	CLB	20	20			7030694	20000	REFERENCE	
0704160-01	A	Apr-16-07 11:14	CLB	20	20						

Comments:	Analyst Initials:
-----------	-------------------

bch_TriMatrix.rpt



SOP Name: Potentiometric pH in Soil and Waste
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Attachment 23.4
Standards Log Report Example

Analytical Standard Record
TriMatrix Laboratories, Inc.

8030282

Description: pH buffer 8.00 Expires: Aug-31-09
Standard Type: Analyte Spike Prepared: Aug-31-07
Solvent: Solvent Lot #1708575 Prepared By: ** Vendor **
Final Volume (mls): 500 Department: Organic - Wet Chemistry
Vials: 2 Last Edit: Jun-12-08 15:29 by BAT

VWR part# BDH5060-500ml
Lot: 1708575
Received 3/07/08

Analyte	CAS Number	Concentration	Units
pH		8	pH Units
Carbon Dioxide, Free		8	pH Units
Carbon Dioxide	124-38-9	8	pH Units
Acidity as CaCO3		8	pH Units
Acidity		8	pH Units

Attachment 23.5
pH Calibration Flowchart



pH Calibration and Quality Control

COPY

Range												LCS		
High							7					10	12	8, 12
Mid				4				7					10	6, 8
Low	2				4				7					2, 6
pH	2	3	4	5	6	7	8	9	10	11	12	pH		

- Run three point curve, ALWAYS starting with 7: High: 7, 10, 12
Slope of the curve must be between 90 - 102%. Mid: 7, 4, 10
Low: 7, 2, 4
- Re-run each point of the curve as a sample as CCVs. Response must be ± 0.05 pH units of true value.
- Run the LCS appropriate to the curve: High: 8, 12
Response must be ± 0.05 pH units of true value. Mid: 6, 8
The LCS are second source buffers. Low: 2, 6
- Every 10 samples and at end of batch run CCVs: High: 10, 12
Response must be ± 0.05 pH units of true value. Mid: 4, 10
The CCVs are the same buffers as the curve. Low: 2, 4

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STANDARD OPERATING PROCEDURE

Chain-of-Custody (COC)

APPROVALS:

QA/QC Manager: *Rich D. Wilbur* Date: 10/27/11
Rich D. Wilbur

QA Officer: *Tom C. Boocher* Date: 10-27-11
Tom C. Boocher

President: *Douglas E. Kriscunas* Date: 10-27-11
Douglas E. Kriscunas

Procedure Number: GR-10-104
 Revision Number: 2.3

Date Initiated: 8/30/96
 Effective Date: 11/10/11

Date Revised: 10/27/11
 Pages Revised: All

By: Tom C. Boocher
 Total Number of Pages: 9

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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 procedure establishes uniform policies for a legally defensible internal chain-of-custody (COC) system for samples requiring defined levels of security.
- 1.2 Following these guidelines will provide unbroken sample tracking from the client source through the analytical process and to final sample disposition.
- 1.3 There are two approaches to maintaining sample custody following receipt by the laboratory and the initial transfer of internal custody (refer to Attachment 6.3 for a flow chart). The two approaches are as follows:
 - 1.3.1 The first approach is used for samples not associated with a project where litigation is expected or likely to be needed, or is requested by the client. In such a case, the project chemist designates in the laboratory information management system (Element) that project samples are handled according to an "external" or general sample custody protocol.
 - 1.3.2 The second approach is used for samples associated with a project where litigation is expected or likely to be needed, or is requested by the client. In such a case, the project chemist designates in the laboratory information management system (Element) that project samples are handled according to an "internal" or specific sample custody protocol.
- 1.4 This procedure also includes general security measures taken to ensure laboratory integrity is maintained in each analytical area.

2.0 DETAILED PROCEDURE

- 2.1 External COC is defined and adhered to as follows:
 - 2.1.1 General building security measures and sample or information access by authorized personnel only must remain in effect at all times.
 - 2.1.2 All laboratory records (paper and electronic), must be maintained in such a way as to minimize physical deterioration, be available and identifiable.
 - 2.1.3 All laboratory records (paper and electronic) must identify the person or persons responsible for information entered which permits the event and chronological reconstruction of TriMatrix Laboratory's involvement with any sample in direct connection with the client report.
 - 2.1.4 Records (both on-site and off-site) include but are not necessarily limited to the following:
 - 2.1.4.1 Sample receipt forms
 - 2.1.4.2 Sample storage information
 - 2.1.4.3 Sample preparation and/or analysis data printouts
 - 2.1.4.4 Data reduction

2.1.4.5 Client correspondence

2.1.4.6 Preliminary and final reports

2.2 Internal COC is defined and adhered to as follows:

2.2.1 In addition to external COC, internal chain-of-custody establishes an intact, continuous record of all physical possession, storage and disposal of sample containers, collected samples, sample aliquots and sample extracts/digestates. Any form on which can be referred to as the "sample".

2.2.2 Internal chain-of-custody must be maintained by one of the following criteria at all times:

2.2.2.1 The sample is in actual physical possession by a laboratory employee.

2.2.2.2 The sample is in constant view of the laboratory employee responsible for physical possession of the sample.

2.2.2.3 The sample is stored in a secure area of the laboratory restricted to authorized personnel only.

2.2.3 An internal COC record must identify all individuals who physically handle a sample. Refer to Attachment 6.1 for an example internal COC form that is automatically generated by the Laboratory Information Management System (Element) when the internal COC is required.

2.2.4 The internal COC record must account for all times and events associated with sample handling and/or movement and must begin at the point established by the applicable federal or state oversight program. Internal COC typically begins at sample collection. However, it may begin at the time clean sample containers are provided by the laboratory.

2.2.5 For projects in which sample containers originate from TriMatrix Laboratories, the internal COC form must accompany containers during transport to the collection site and be returned when collected samples are returned to the laboratory.

2.2.6 Shipping cooler custody seals and/or individual sample containers (if affixed by the client) are to be submitted. Receipt of a cooler without an intact seal must be documented on the sample receipt checklist.

2.2.7 Sample login staff are responsible for maintaining sample storage areas and related receipt documentation which are part of an internal COC project. In the event a sample login technician is not available when samples arrive (such as, after normal business hours), the following persons must assume responsibility for initiating internal COC:

2.2.7.1 Any Laboratory analyst or technician

2.2.7.2 Any Project chemist

2.3 A COC sequence of events is as follows:

- 2.3.1 Upon receipt and proper acknowledgment of coolers containing samples designated for internal COC, sample technicians must perform a thorough inspection of any and/or all custody seals. Should any seal arrive damaged, the project chemist and/or client must be immediately notified.
- 2.3.2 All paperwork accompanying sample coolers must be retrieved and checked for completeness and accuracy. Any discrepancies between received samples and the paperwork or any project information must be noted and recorded. Inform the project chemist of all COC discrepancies. Document the discrepancy and take or suggest all appropriate corrective action to minimize a repeat discrepancy.
- 2.3.3 Only when samples have been properly accounted for and any discrepancies resolved may they be logged into the laboratory information management system (Element). During log-in, the option to designate samples as internal COC must be selected. This initiates the creation of the COC form for each analysis sample (Attachment 6.1) which must be signed with each change in location and/or custody.
- 2.3.4 After log-in, samples must be moved to a designated storage area (Attachment 6.2). Access to all sample storage areas is limited to employees only for security reasons.
- 2.3.5 Chain-of-custody forms are placed in folders located in the laboratory by the walk-in cooler. The technician logging in samples must ensure each sample has the appropriate type and number of sign-out sheets.
- 2.3.6 Samples requiring internal COC status are communicated to laboratory areas through arrival logs, worklists and worksheets. These forms are generated by Element. Area supervisors and group leaders must ensure all chemists and technicians are aware of any and all internal COC samples. The signing of COC forms must be strictly and consistently done. Any technician or analyst not completing the COC form will be reprimanded accordingly.
- 2.3.7 When preparing to analyze internal COC samples, the COC form must be completely filled in with the removal time, analyst's name and the return time.
- 2.3.7.1 Internal COC forms must be filled in each time samples are removed from a designated storage area.
- 2.3.7.2 Interim sample storage areas are located in each laboratory area for use on in-process samples which also limit access to employees only. After signing for an internal COC sample to remove from a designated storage area, samples may be stored in these areas until analysis is complete.
- 2.3.7.3 Once analysis is complete, samples must be returned to the original designated storage area. The internal COC form must be completed before the analyst may proceed to any other task.
- 2.3.8 Once a project submittal has been completed by the laboratory, the project chemist must review the COC folder contents by checking for COC form completeness. After reviewing, include the completed COC paperwork in the project folder.
- 2.4 The transfer of samples to an approved subcontract laboratory for analysis must adhere to the same COC requirements specified for the project.

3.0 REPORTING AND DELIVERABLES

- 3.1 The completed internal chain-of-custody form must accompany the client report unless otherwise specified.

4.0 QUALITY ASSURANCE

- 4.1 All laboratory quality assurance activities as outlined in the TriMatrix Quality Assurance Manual and as specified in any related Standard Operating Procedure must be adhered to.
- 4.2 The laboratory is a restricted access area. Only authorized personnel are allowed into the laboratory without an escort. Visitors must sign the Visitor Log book when entering and exiting the laboratory.
- 4.3 Project chemists are responsible for the following with respect to COC communications:
- 4.3.1 Communicating COC sample information to sample technicians and/or analysts as soon as this knowledge is available.
 - 4.3.2 Monitoring sample submittal progress.
 - 4.3.3 Checking each COC form for completeness.
- 4.4 Sample technicians are responsible for receiving, inspecting and logging in the samples correctly for COC status, and for printing and placing COC sign-out forms into folders.
- 4.5 Chemists and analysts must follow a strict protocol when performing analyses on COC samples. Each time a sample is removed from a designated storage area, the COC form must be signed with the following information:
- 4.5.1 Removal date and time
 - 4.5.2 Return date and time
 - 4.5.3 Name and signature
- 4.6 Area supervisors and group leaders are responsible for mandating that area chemists follow the internal COC protocol.

5.0 REFERENCES

- 5.1 TriMatrix Laboratories Quality Assurance Manual, latest revision.

6.0 ATTACHMENTS

- 6.1 Internal Chain-of-Custody Form Example

- 6.2 Facility Security Layout
- 6.3 Internal Chain-of-Custody Flowchart

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SOP Name: Chain-of-Custody (COC)

Revision Number: 2.3
Date Revised: 10/27/11
Date Initiated: 8/30/96

SOP Number: GR-10-104

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Attachment 6.1
Internal Chain-of-Custody Form Example

TriMatrix Laboratories, Inc.
Internal Chain of Custody

1110370

Client: [REDACTED] Received: Oct-19-11 16:00:00
Project: IPP Monthly Monitoring / Water Quality Received By: Donna M. Nardin
Number: 29383 Temp (°C): 1.9

Printed: 10/27/2011 1:27:13PM

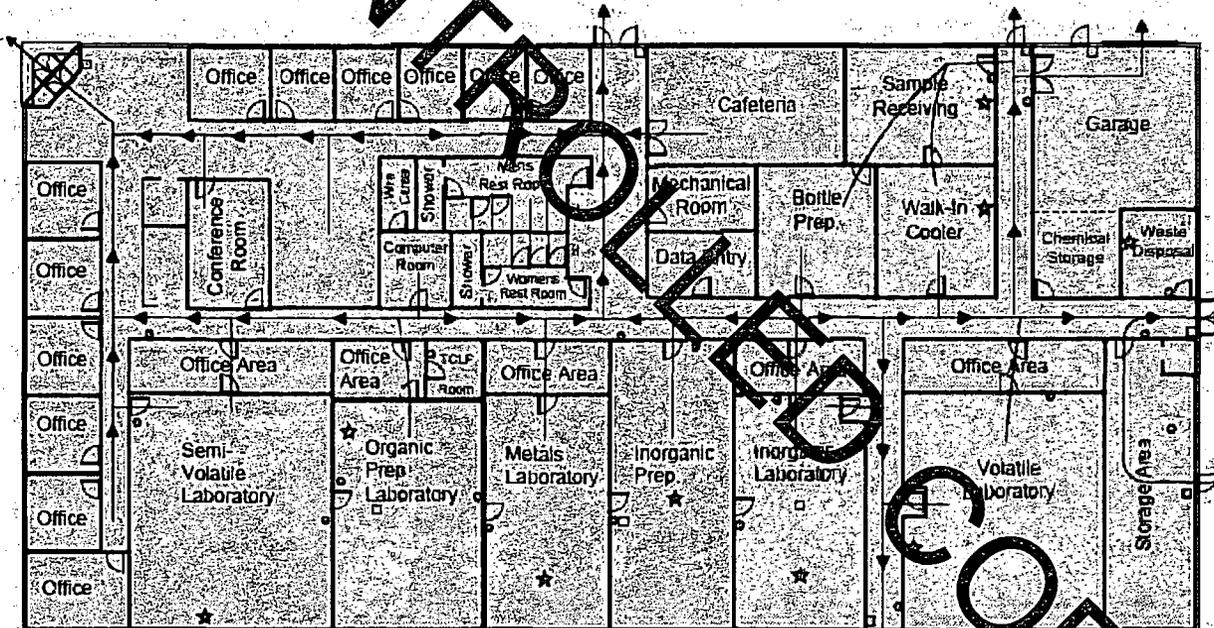
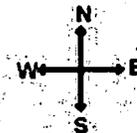
1110370-02 (West Outfall Monthly Composite) Sampled Oct-19-11 13:00:00

1110370-02 A [02_1000mL Amber Glas Semivolatiles GC	Out Oct-21-11 15:52:03 by SKA	In Jan-01-80 00:00:00 by
1110370-02 A 01 [SVGC Extract] Semivolatiles GC	Out Oct-21-11 15:52:24 by SKA	In Jan-01-80 00:00:00 by
1110370-02 F 01 [04_0500mL Plastic p Inorganic - Wet Chemistry	Out Oct-20-11 08:28:59 by CLD	In Jan-01-80 00:00:00 by
1110370-02 G 01 [Metals Extract] Metals	Out Oct-20-11 10:24:56 by RDB	In Jan-01-80 00:00:00 by
1110370-02 G 02 [Metals Extract] Metals	Out Oct-25-11 12:56:11 by MAS	In Jan-01-80 00:00:00 by

Attachment 6.2
Facility Security Layout



Laboratory Evacuation Diagram

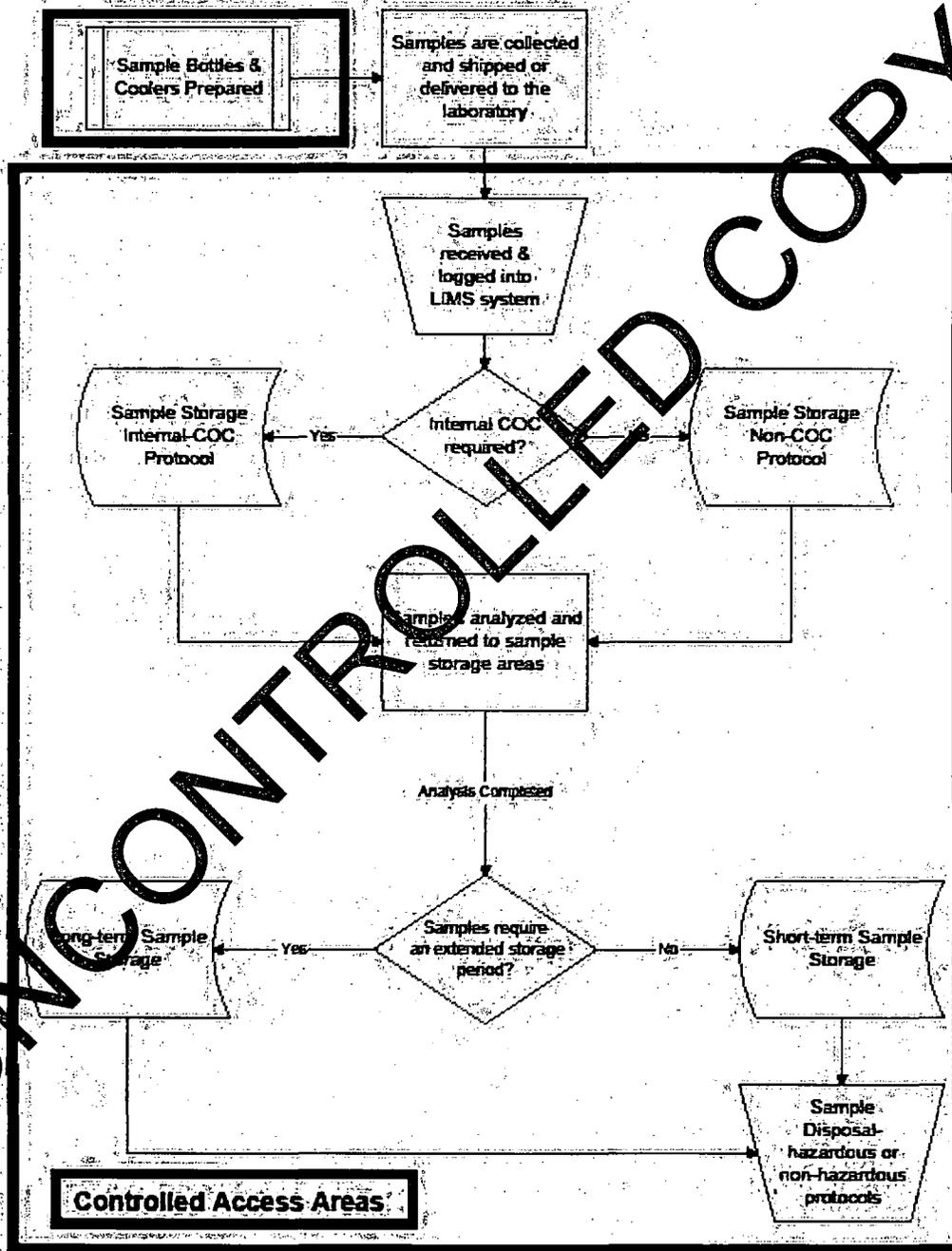


* Meeting area is in the front (North) parking lot

- Fire Extinguisher
- ⊕ First Aid Kit
- ☐ Safety Shower
- ★ Eyewash Station
- ⊞ Pull Station

Revision 1.1

**Attachment 6.3
Internal Chain-of-Custody Flowchart**





STANDARD OPERATING PROCEDURE

Sample Receipt and Log-in

APPROVALS:

Area Supervisor: _____

[Signature]
Gary L. Wood

Date: 1-12-12

QA Officer: _____

[Signature]
Tommy Bostler

Date: 1-12-12

Laboratory President: _____

[Signature]
Douglas E. Kriscunas

Date: 1-12-2012

Procedure Number: GR-15-100
Revision Number: 3.3

Date Initiated: 2/27/96
Effective Date: 1/25/12

Date Revised: 1/12/12
Pages Revised: All

By: Rick D. Wilburn

Total Number of Pages: 32

UNCONTROLLED COPY

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 Saturday. Sample receipt at other times must be arranged through a project chemist or the log-in staff.
- 1.3 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 at 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 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

- 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.

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

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 when 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.

2.4 Step 3

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, RT 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 24 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.

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
- 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 Volatile 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 bench sheets 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 (if 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-15-100. 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 ₂ SO ₄	TKN, Nitrate-Nitrite, Phosphorus, COD, Ammonia
13	Brown	H ₂ SO 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 $\pm 0.5^\circ\text{C}$ of the cooler thermometer. Document the test results in the Infra-Red Thermometer Calibration Record logbook (Attachment 6.13). A difference of greater than $\pm 0.5^\circ\text{C}$ 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 submitter 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 Performance 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.
- 4.14 TriMatrix reserves the right to refuse or reject a sample for a variety of reasons. The reasons fall into two basic categories; health based, and quality based.
 - 4.14.1 Health based reasons are of immediate concern. Samples that may pose a risk or become unsuitable for handling, transport, or processing, for any health, safety, environmental or other reason, whether or not due to the presence in the sample of any hazardous substance and whether or not such presence has been disclosed to TriMatrix will immediately be rejected.
 - 4.14.2 Quality based concerns are more common reasons for sample rejection. Items such as incomplete sample identification, inappropriate sample container, inadequate sample volume, broken container, and hold time exceedences, may also result in sample rejection. The most common quality based violations are detailed on Attachment 6.5, "Sample Receiving Non-Conformance Report".
 - 4.14.3 All items flagged on the non-conformance report will be evaluated by the project manager. The project manager will discuss all violations with the client and samples will rejected or qualified accordingly.

5.0 REFERENCES

- 5.1 TriMatrix SOP GR-15-100 *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
- 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

UNCONTROLLED COPY

Attachment 6.1
Sample Receipt Record
Sample Receipt Record


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			Delivery Method Letter	Submittal Number (Project Chemist)	Folder Prepared (Log-In ✓)
			Time	AM	PM			
1-1								
1-2								
1-3								
1-4								
1-5								
1-6								
1-7								
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Attachment 6.3
Sample Receiving Log-In Checklist (page 2)

SAMPLE RECEIVING / LOG-IN CHECKLIST - page 2

Project Chemist Use	Log-In Use
Notify Laboratory Personnel of Short Hold-Times and/or Rush Work <input type="checkbox"/> NONE (Lab personnel notified/date) _____ <input type="checkbox"/> Inorganics _____ <input type="checkbox"/> Microbiology (bacteria) _____ <input type="checkbox"/> Metals Prep _____ <input type="checkbox"/> Metals _____ <input type="checkbox"/> GC-Volatiles _____ <input type="checkbox"/> MS-Volatiles _____ <input type="checkbox"/> Semi-Vol. Prep _____ <input type="checkbox"/> GC-Semi-Volatiles _____ <input type="checkbox"/> MS-Semi-Volatiles _____	Log Samples into LIMS Sample No. _____ N/A Yes <input type="checkbox"/> Receive samples in LIMS <input type="checkbox"/> Date/Time received entered in LIMS <input type="checkbox"/> Read project and submittal narratives <input type="checkbox"/> Enter VOC rack/tray number into submittal narrative <input type="checkbox"/> Enter sample information in LIMS <input type="checkbox"/> Add any sample narratives <input type="checkbox"/> If non-conformance issues, add sample qualifiers <input type="checkbox"/> Print sample number labels
Log-In Priority <input type="checkbox"/> RUSH <input type="checkbox"/> Standard Project Chemist Notes to Log-In Personnel Trip Blank: <input type="checkbox"/> Log-in <input type="checkbox"/> Do not log-in <input type="checkbox"/> Prep Storage Blank for Client (VOCs) <input type="checkbox"/> Sub-Contracting required <input type="checkbox"/> Coolant required <input type="checkbox"/> Non-TriMatrix or non-standard container type(s) received Check pH of container type _____ Expected pH: _____ <input type="checkbox"/> Adjust if necessary <input type="checkbox"/> Adjust pH of orange-tagged containers <input type="checkbox"/> Lab-filter samples and document on Preservation Form	Log-In Analyst (initials/date/time) _____ Label Sample Containers N/A Yes No <input type="checkbox"/> LIMS tag includes tag? <input type="checkbox"/> DISCREPANCIES CORRECTED IN LIMS Initials/Date: _____ <input type="checkbox"/> Applicable stickers applied to labels? <input type="checkbox"/> MS/MSD sample <input type="checkbox"/> Composite before analysis <input type="checkbox"/> Applicable stickers applied to containers? <input type="checkbox"/> Waste sample <input type="checkbox"/> PT sample <input type="checkbox"/> USDA regulated <input type="checkbox"/> Orange-tagged containers present? <input type="checkbox"/> Adjust pH per Project Chemist <input type="checkbox"/> Initials and Date/Time Adjusted on orange tag? <input type="checkbox"/> Initials and Date/Time Adjusted on Preservation Form? Verify Label Accuracy <input type="checkbox"/> Second analyst checked labels for accuracy? <input type="checkbox"/> Verify that Orange-tagged containers adjusted/initialed?
	Labeled by (initials/date) _____ Verified by (initials/date) _____
	Sample Storage Check all that apply bacteria..... <input type="checkbox"/> bacteria refrigerator non-volatiles..... <input type="checkbox"/> walk-in cooler volatiles..... <input type="checkbox"/> volatile lab refrigerator waste..... <input type="checkbox"/> waste cabinet waste VOCs..... <input type="checkbox"/> log-in hood refrigerator low-level Hg <input type="checkbox"/> metals lab - DO NOT STORE IN WALK-IN
Sample Narratives to be added at Log-in _____ _____ _____ _____	Paperwork N/A Yes <input type="checkbox"/> original COC (white) <input type="checkbox"/> copy of COC (yellow) <input type="checkbox"/> receiving/log-in checklist <input type="checkbox"/> additional cooler information form <input type="checkbox"/> sample preservation verification <input type="checkbox"/> sample receiving non-conformance form <input type="checkbox"/> shipping documents <input type="checkbox"/> custody seals <input type="checkbox"/> arrival log <input type="checkbox"/> other (note)

**Attachment 6.4
Sample Receiving/Log-In Checklist
Additional Cooler Information**



**SAMPLE RECEIVING / LOG-IN CHECKLIST
ADDITIONAL COOLER INFORMATION**

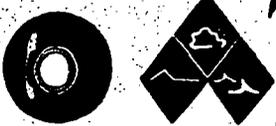
Recorded by (initials/date)		Client		Project-Subtotal No.	
Receipt Log No.		Sample No.		Project Chemistry	

<table border="1" style="width:100%; border-collapse: collapse;"> <tr><td>Cooler No.</td><td>Time</td></tr> <tr><td colspan="2">Custody Seals</td></tr> <tr><td><input type="checkbox"/> none</td><td></td></tr> <tr><td><input type="checkbox"/> present / intact</td><td></td></tr> <tr><td><input type="checkbox"/> present / not intact</td><td></td></tr> <tr><td colspan="2">Coolant Location:</td></tr> <tr><td colspan="2">Dispersed / Top / Middle / Bottom</td></tr> <tr><td colspan="2">Coolant / Temperature Taken Via:</td></tr> <tr><td><input type="checkbox"/> loose ice / avg 2-3 containers</td><td></td></tr> <tr><td><input type="checkbox"/> bagged ice / avg 2-3 containers</td><td></td></tr> <tr><td><input type="checkbox"/> blue ice / avg 2-3 containers</td><td></td></tr> <tr><td><input checked="" type="checkbox"/> none / avg 2-3 containers</td><td></td></tr> <tr><td colspan="2">Alternate Temperature Taken Via:</td></tr> <tr><td><input type="checkbox"/> temperature blank (tb)</td><td></td></tr> <tr><td><input type="checkbox"/> 1 container</td><td></td></tr> <tr><td>Recorded °C</td><td>Correction Factor °C</td><td>Actual °C</td></tr> <tr><td>tb</td><td></td><td></td></tr> <tr><td colspan="3">tb location: representative / in ice</td></tr> <tr><td>1</td><td></td><td></td></tr> <tr><td>2</td><td></td><td></td></tr> <tr><td>3</td><td></td><td></td></tr> <tr><td colspan="3">Average °C</td></tr> <tr><td colspan="3"><input type="checkbox"/> Cooler ID on COC?</td></tr> <tr><td colspan="3"><input type="checkbox"/> VOC trip blank received?</td></tr> </table>	Cooler No.	Time	Custody Seals		<input type="checkbox"/> none		<input type="checkbox"/> present / intact		<input type="checkbox"/> present / not intact		Coolant Location:		Dispersed / Top / Middle / Bottom		Coolant / Temperature Taken Via:		<input type="checkbox"/> loose ice / avg 2-3 containers		<input type="checkbox"/> bagged ice / avg 2-3 containers		<input type="checkbox"/> blue ice / avg 2-3 containers		<input checked="" type="checkbox"/> none / avg 2-3 containers		Alternate Temperature Taken Via:		<input type="checkbox"/> temperature blank (tb)		<input type="checkbox"/> 1 container		Recorded °C	Correction Factor °C	Actual °C	tb			tb location: representative / in ice			1			2			3			Average °C			<input type="checkbox"/> Cooler ID on COC?			<input type="checkbox"/> VOC trip blank received?			<table border="1" style="width:100%; 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**Attachment 6.6
TriMatrix Sample Container Tag and Log-In Labels**

381536	
WG-011305- ML-011	Office
PROJ: _____	_____
SUBM: January 13, 2005 Samples	
F=0	R=0 C=0 H=0

**Project Specific Sample
MS/MSD**



**TriMatrix
Laboratories, Inc.**

**USDA REGULATED
SOIL SAMPLE**

Client _____	PINK
Project Number: _____	
Date: _____ Time: _____	
Preservative: <u>H₂S</u> _____	
Sampled By: _____	
Sample Location: _____	

Field pH _____
Please verify pH in laboratory.

Initials Date

**Store at
Room Temperature**

**Composite
Before Analysis**

PE SAMPLE
**do not dispose of
until notified**

**Composite
Before Analysis**

 **Waste Sample**
Return to appropriate
shelf in waste cabinet

Attachment 6.7
Sample Preservation Verification Form

SAMPLE PRESERVATION VERIFICATION FORM

page ___ of ___

Client				Project-Substance No.		
Receipt Log No.		Completed By (initials/dates)		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 ₂ SO ₄	H ₂ SO 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 ₂ SO ₄	H ₂ SO 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						

Container Size (mL)	Original Vol. of Preservative (mL)
Container Type 5: NaOH	
500	2.5
1000	5.0
Container Type 4: H ₂ SO ₄	
125	0.5
250	1.0
500	2.0
1000	4.0
Container Type 13: H ₂ SO ₄	
500	2.5

COPY

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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, 4, and 15.

Attachment 6.8
Out-of-Control Sample pH Tag

Out-of-Control Sample pH		
Initial pH	Final pH	mL Added
OK <input type="checkbox"/>	Still Out <input type="checkbox"/>	

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**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	7 Hours
Heterotrophic Plate Count - Non-Potable Water for Non-Compliance Purposes	24 Hours
Chromium, Hexavalent	4 Hours
BOD	48 Hours
Color	48 Hours
MBA's / 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

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Attachment 6.10
Container Packing List (page 1)



Container Packing List

For any questions regarding these containers, contact a Project Chemist at (616) 975-4500.

Client: _____ Project: _____ Page 1 of _____

#	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
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18																													
19																													
20																													
Total Containers																													

This container type requires field-filtering.

MATRIX	#	TEST	SIZE	TYPE	QUANTITY	OPTIONS	PRESERVATIVE	TAG COLOR
WATER	0	Unpreserved Purgable Organics	40 mL	Clear Glass Vial	40		Cool to 4° C	Yellow & Black Stripe
	1	Preserved Purgable Organics	40 mL	Clear Glass Vial (pre-preserved)	40		HCl; Cool to 4° C	Yellow
	2	Non-Purgable Organics	100 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 ₂ SO 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	1000/500, 1000/500		pH <2 w/ H ₂ SO ₄	Dark Blue
	8	Bacteria		125 mL Plastic (pre-preserved)	125		Na ₂ S ₂ O ₅ ; 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 ₂ SO ₄	Light Blue
	11	TOC		40 mL Amber Vial	40		pH <2 w/ H ₂ SO ₄	Pink
	12	DRO		1000 mL Amber Glass	1000		pH <2 w/ HCl	Gray
SOLIDS	13	% Solids	500 mL Amber Glass	500		pH <2 w/ H ₂ SO 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
	16	Inorganic Metals		WM Plastic	125, 250, 500, 1000		Cool to 4° C	White
	17	Purgable Organics		WM Clear Glass	125, 250, 500, 1000		Cool to 4° C	Manila
MISC	18	Purgable Organics - Bulk	60 mL	WM Clear Glass	60		Cool to 4° C	Light Yellow
	19	TCVP 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	Purgable Organics		Excure Sampler	5g, 25g		Cool to 4° C	Label on Bag
	22	Purgable 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)
23								
24								
25	Pesticide WVs 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		

Attachment 6.10 Container Packing List (page 2)



Project Chemist Initials:	Added to Calendar & Folders (initials/date):	Revision: 3	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	<input type="checkbox"/> Jan	<input type="checkbox"/> Feb	<input type="checkbox"/> Mar	<input type="checkbox"/> Apr	<input type="checkbox"/> May	<input type="checkbox"/> Jun
	<input type="checkbox"/> Jul	<input type="checkbox"/> Aug	<input type="checkbox"/> Sep	<input type="checkbox"/> Oct	<input checked="" type="checkbox"/> Nov	<input type="checkbox"/> Dec
Weeks	<input type="checkbox"/> 1	<input type="checkbox"/> 2	<input type="checkbox"/> 3	<input type="checkbox"/> 4	<input type="checkbox"/> 5	<input type="checkbox"/> 6
Days	<input type="checkbox"/> M	<input type="checkbox"/> T	<input type="checkbox"/> W	<input type="checkbox"/> TH	<input type="checkbox"/> F	<input type="checkbox"/> S

Containers will be Picked Up or Shipped via: First Overnight Standard Overnight
 Priority Overnight Express Saver
 2 Day Ground
 Saturday Delivery TriMatrix Courier
 Other: _____

Telephone No: _____
 Shipment to be billed to FedEx Account No.: _____

Shipment to include: COCs (Qty) _____ Custody Seals Temperature Blanks
 SDS Sheets for all preservatives used WB TM#? Y N
 Cooler Banding Required

Comments: _____

Assembled by/Date:	Checked by/Date:	Shipped by/Date:																														
<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 10%;">Coolers Sealed With</th> <th style="width: 10%;">Tape</th> <th style="width: 10%;">Banding Strap</th> </tr> <tr> <td>Used:</td> <td><input type="checkbox"/></td> <td><input type="checkbox"/></td> </tr> <tr> <td></td> <td><input type="checkbox"/></td> <td><input type="checkbox"/></td> </tr> </table>	Coolers Sealed With	Tape	Banding Strap	Used:	<input type="checkbox"/>	<input type="checkbox"/>		<input type="checkbox"/>	<input type="checkbox"/>	Tracking Number Label(s): _____ _____ _____																						
Coolers Sealed With	Tape	Banding Strap																														
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STANDARD OPERATING PROCEDURE

Setaflash Closed-Cup Flashpoint

SW-846 Method 1020A

APPROVALS:

Area Supervisor: *Heather L. Brady* Date: 4-11-13
Heather L. Brady

QA Officer: *Tom C. Boocher* Date: 4-12-13
Tom C. Boocher

Laboratory President: *Douglas E. Kriscunas* Date: 4/11/13
Douglas E. Kriscunas

Procedure Number: GR-18-124
Revision Number: 1.5

Date Initiated: 10/1/99
Effective Date: 4/15/13

Date Revised: 4/4/13
Pages Revised: All

By: Tom C. Boocher
Total Number of Pages: 11

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
_____	_____	_____
_____	_____	_____
_____	_____	_____

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1.0 SCOPE AND APPLICATION

- 1.1 This procedure is applicable to the flashpoint of liquid waste samples. The procedure, while not technically applicable, is also used to determine the flashpoint of solid waste samples. Flashpoints reported from the analysis of solid waste samples will be qualified.
- 1.2 This procedure is not applicable to waste samples that are difficult to homogenize where a representative aliquot cannot be analyzed in the tester.
- 1.3 The applicable range is 68 - 200° F.
- 1.4 The minimum flashpoint temperature reported for this analysis is 68° F.

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 1020A, "Setaflash Closed-Cup Method for Determining Ignitability"*

3.0 SUMMARY OF PROCEDURE

- 3.1 A small sample aliquot is heated at a low, constant rate while a flame is intermittently directed into the cup at regular intervals to check for a vapor flash.
- 3.2 The flashpoint is the lowest temperature at which application of the flame ignites vapors above the sample and a flash is observed.

4.0 PARAMETER OR COMPOUND LIST

- 4.1 Flashpoint

5.0 REFERENCED SOPs

- 5.1 Trimatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

- 6.1 Use the actual ambient air pressure of the laboratory at the time of testing to calculate adjusted flashpoint.
- 6.2 Solid samples must be homogeneous and representative to give an accurate result. If not, narrate that a representative and/or homogenous sample could not be evaluated.
- 6.3 Do not open containers unnecessarily or make sample transfers at a temperature within 30° F of the flashpoint. Do not mix solid samples excessively to minimize the loss of volatile materials.

- 6.4 Use the draft-free box during the flashpoint determination to minimize drafts across the flashpoint tester.
- 6.5 Care must be taken to assure that all test conditions are consistent. These conditions include the size of the flame, the rate of temperature increase, the frequency of dipping the flame into the sample vapor and observation of the flash itself.

7.0 SAFETY PRECAUTIONS

- 7.1 Wear a laboratory coat and approved safety glasses while in the 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-18-102.
- 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 exposure 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 Waste samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-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 Collect and store samples in glass bottles with PTFE-lined lids.
- 8.2 Transport and store samples at 0 - 6° C until the time of analysis.
- 8.3 Shake liquid waste samples just prior to removing a sample aliquot.
- 8.4 Do not mix and/or homogenize solid waste samples unless it can be done quickly within the sample container without the risk of losing entrained volatiles. Test at least two representative aliquots from within the sample.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Koehler Rapid Tester Model K16502, Closed-cup

9.2 ASTM Thermometer No. 9F Range 20 – 230° F, 287 mm

9.3 Barometer, verified annually

9.4 Syringe, 2 ±0.1 mL capacity

Note: Test the syringe for accuracy using an analytical balance and water. Maintain a record of the testing.

10.0 ROUTINE PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

10.1 Thoroughly clean and dry all parts of the flashpoint tester cup between samples. Additional cleaning when necessary can be accomplished with solvents such as methylene chloride which are capable of removing oily and stubborn material.

10.2 Visually inspect the flashpoint tester prior to use to insure proper mechanical functionality.

11.0 CHEMICALS AND REAGENTS

11.1 Reference standard, p-xylene, 99%+, anhydrous

Note: Do not store p-xylene in the hood. Store in a flammables cabinet.

12.0 STANDARDS PREPARATION

12.1 There is no standard preparation directly associated with this procedure.

13.0 SAMPLE PREPARATION

13.1 No sample aliquot shall be placed in the tester cup that exceeds 30° F below the sample's expected flashpoint.

14.0 CALIBRATION PROCEDURES

14.1 Use the thermometer reading, not the LED readout for the temperature of the testing unit.

14.2 Determine the p-xylene flashpoint by adding 2.0 mL (using provided syringe) through the entry port and determining the flashpoint based on the procedure. Repeat. When the unit is properly operating, the average flashpoint will be 81 ±1.5° F.

14.3 If the value lies outside this window, verify all operating conditions and repeat the test. If the average is still outside the acceptance window, contact the area supervisor.

14.4 Do not analyze samples unless an acceptable p-xylene test can be made.

15.0 ANALYTICAL PROCEDURE

- 15.1 Thoroughly inspect, clean and dry all parts of the tester cup and cover assembly before testing a sample. Be sure to remove all cleaning solvent.
- 15.2 Check to see that the tester temperature is below 68° F. Add 2.0 mL of liquid sample through the injection port with the syringe. For solids, use approximately 2 g of sample.
- 15.3 For the first determination, switch the tester on and adjust the target temperature to ramp to by depressing the red preset button while turning the black knob. This is helpful for checking the calibration and when the flashpoint temperature is approximately known. Otherwise, set to 210° F. Light the flame.
- 15.4 Allow the temperature to reach 68° F then check for a flash by applying the flame for a second.
- 15.4.1 If a flash is observed as evidenced by a large blue flame appearing and propagating itself across the surface of the sample, report the flashpoint as less than 68° F.
- Note: Do NOT confuse the bluish halo that sometimes surrounds the test flame with a true flashpoint.
- 15.4.2 If no flash is observed, allow the temperature to rise 9° F and check again.
- 15.4.3 Repeat the test flame every 9° F until a flash is observed or until 210° F is reached.
- 15.4.4 If a flash is not observed at 210° F, report the flashpoint as greater than 200° F.
- 15.5 Allow the tester to cool, clean the cup then inject a new sample aliquot. Do NOT retest the first sample aliquot.
- 15.6 Bring the tester to the temperature of the last interval before the flash was observed then test for a flash every 1° F until a flash is observed.
- 15.7 Record this last observation as the observed sample flashpoint.
- 15.8 When enough sample is available, repeat the 1° F determination with a fresh aliquot. The result of the duplicate analysis must be within 3° F of the initial analysis. If this criterion is not met, qualify the result as estimated.

16.0 CALCULATIONS AND DATA HANDLING

- 16.1 Observe and record the ambient barometric pressure at the time of the test in mm of mercury. When the pressure differs from 760 mm, correct the flashpoint as follows:

$$[F + [0.06 (760 - P)]] = \text{Corrected Flashpoint}$$

Where:

F = observed flashpoint, ° F
P = ambient barometric pressure, mm mercury

Note: The corrected flashpoint needs to be reported only if the corrected flashpoint is greater than 1° F from the observed flashpoint.

- 16.2 Report the first 1° F result if a flashpoint is observed between 68° F and 200° F. Otherwise, report <68° F or >200° F as appropriate.

17.0 DATA REPORTING AND DELIVERABLES

- 17.1 Record all measurements made.
- 17.2 All solid sample flashpoints must be qualified as follows:

"SW-846 method 1020 is not designed to be used for solid waste matrices. The reported flashpoint should not be used to determine the hazardous waste characteristic of ignitability"

18.0 QUALITY ASSURANCE

- 18.1 Run each sample in duplicate using the 5 F frequency test. The duplicate must be within 3° F of the first result. If this criterion is not met, repeat the duplicate once to determine which result to report. If still not within 3° F of the first result or the first duplicate, qualify the result as estimated.
- 18.2 A p-xylene standard (SCV) from a source other than the accuracy check standard must be analyzed in duplicate with each batch of up to 20 samples. The average value must be $81 \pm 1.5^\circ$ F. If this criterion is not met, all samples analyzed in the batch must be re-analyzed or qualified as estimated.
- 18.3 Notify the area supervisor if the second-source p-xylene standard fails. Perform the SCV analysis at the beginning of the analytical batch.
- 18.3 All flashpoint thermometers must be calibrated against a NIST-certified thermometer prior to their initial use and thereafter on an annual basis.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

- 19.1 Before the analysis of 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 Analyze four 2.0 mL aliquots of p-xylene. The average value must be $81 \pm 1.5^\circ$ F.

- 19.1.1.2 Input results to the IDC spreadsheet located on the laboratory intranet library.
- 19.1.1.3 If the acceptance window is not met, locate and correct the source of the problem and repeat the study.
- 19.1.1.4 Repeated failure will confirm a general problem with the procedure. If this occurs, locate and correct the source of the problem and repeat the study.
- 19.1.1.5 Samples may not be analyzed by any analyst or on any instrument until certification demonstration of capability study has been successfully completed.
- 19.1.1.6 Give a copy of all IDC spreadsheets and raw data to the Quality Assurance department.
- 19.1.2 Continuing Demonstration of Capability (CDC)
- 19.1.2.1 A CDC must be performed annually.
- 19.1.2.2 The CDC may be accomplished by repeating the IDC study, by using four consecutive SCV results obtained from routine sample analysis or by running an acceptable PT sample.
- 19.1.2.3 Give a copy of all CDC spreadsheets and raw data to the Quality Assurance department.
- 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 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. These documents 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

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 1, July, 1992, Method 1020A, "Setafash Closed-Cup Method for Determining Ignitability"*

23.0 ATTACHMENTS

- 23.1 Preparation Batch Report Example
23.2 Analysis Sequence Report Example
23.3 Data Review Report Example

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Attachment 23.1
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH **0800584** Page 1 of 1

Printed: 12/16/2008 3:26:18PM

Inorganic - Wet Chemistry, Waste, Method-Specific Preparation
(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0801032	FlashPoint 1020A	0801059	FlashPoint 1020A	0801108	FlashPoint 1020A
0801156	FlashPoint 1020A	0801158	FlashPoint 1020A	0801159	FlashPoint 1020A
0801218	FlashPoint 1020A				

Lab Number	Contain	Prepared	By	Initial (g)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client/ QC Type	Extraction Comments
0800584-BLK1		Jan-17-08 08:01	INR	2	2					BLANK	
0800584-DUP1		Jan-17-08 08:01	INR	2	2		0801032-02			DUPLICATE	
0800584-BS1		Jan-17-08 08:01	INR	2	2			71202	100	CS	
0801032-02	C	Jan-17-08 08:01	INR	2	2					[REDACTED]	
0801059-01	A	Jan-17-08 08:01	INR	2	2					[REDACTED]	if there are VOCs, they get sample 1st
0801059-02	A	Jan-17-08 08:01	INR	2	2					[REDACTED]	if there are VOCs, they get sample 1st
0801108-01	B	Jan-17-08 08:01	INR	2	2					[REDACTED]	
0801156-01	A	Jan-17-08 08:01	INR	2	2					[REDACTED]	if there are VOCs, they get sample 1st
0801158-01	A	Jan-17-08 08:01	INR	2	2					[REDACTED]	if there are VOCs, they get sample 1st
0801158-02	A	Jan-17-08 08:01	INR	2	2					[REDACTED]	if there are VOCs, they get sample 1st
0801159-01	A	Jan-17-08 08:01	INR	2	2					[REDACTED]	if there are VOCs, they get sample 1st
0801218-01	A	Jan-17-08 08:01	INR	2	2					[REDACTED]	



SOP Name: Setafash Closed-Cup Flashpoint
 SW-846 Method 1020A
 SOP Number: GR-18-124

Revision Number: 1.5
 Date Revised: 4/4/13
 Date Initiated: 10/01/99

**Attachment 23.2
 Analysis Sequence Report Example**

TriMatrix Laboratories, Inc. ANALYSIS SEQUENCE **8011708** Page 1 of 1

Printed: 12/16/2008 3:29:52PM

Inorganic - Wet Chemistry, Waste, Jan-17-08
 Instrument = 186, Calibration = UNASSIGNED

Sequence Analyses:
 FlashPoint 1020A

Lab Number	Analysis	Corr	STD ID	ISTD ID	Client / QC Type	Extraction Comments
0801032-02	FlashPoint 1020A	C			[REDACTED]	
0800584-DUP1	QC				DUPLICATE	
0801059-01	FlashPoint 1020A	A			[REDACTED]	if there are VOCs, they get sample 1st
0801059-02	FlashPoint 1020A	A			[REDACTED]	if there are VOCs, they get sample 1st
0801108-01	FlashPoint 1020A	B			[REDACTED]	
0801156-01	FlashPoint 1020A	A			[REDACTED]	if there are VOCs, they get sample 1st
0801158-01	FlashPoint 1020A	A			[REDACTED]	if there are VOCs, they get sample 1st
0801158-02	FlashPoint 1020A	A			[REDACTED]	if there are VOCs, they get sample 1st
0801159-01	FlashPoint 1020A	A			[REDACTED]	if there are VOCs, they get sample 1st
0801218-01	FlashPoint 1020A	A			[REDACTED]	
0800584-BS1	QC				LCS	
8011708-SCV1	QC		7120233		SECONDARY CAL CHECK	
0800584-BLK1	QC				BLANK	

**Attachment 23.3
 Data Review Report Example**

TriMatrix Laboratories, Inc.

Data Review Report -- Wet Chem Lab
 Sequence = 8011708 Page 1 of 1

on 12/16/2008 at 3:33

<u>SampleID</u>	<u>Analysis</u>	<u>IResult</u>	<u>Diln</u>	<u>Result</u>	<u>FMRL</u>	<u>Qualifier</u>	<u>Recovery</u>	<u>RPD</u>	<u>Analyzed</u>	<u>Analyst</u>	<u>Batch</u>
0801032-02	FlashPoint 1020A	999	1	999	68	GN014,W C015			1/17/2008 1:01	INR	0800584
0800584-DUP1	FlashPoint 1020A	999	1	999	68	WC015		0	1/17/2008 1:01	INR	0800584
0801059-01	FlashPoint 1020A	999	1	999	68	GN014,W C015			1/17/2008 1:01	INR	0800584
0801059-02	FlashPoint 1020A	999	1	999	68	GN014 C015			1/17/2008 1:01	INR	0800584
0801108-01	FlashPoint 1020A	999	1	999	68	GN014 C015			1/17/2008 1:01	INR	0800584
0801156-01	FlashPoint 1020A	999	1	999	68	WC015			1/17/2008 1:01	INR	0800584
0801158-01	FlashPoint 1020A	171	1	171	68				1/17/2008 1:01	INR	0800584
0801158-02	FlashPoint 1020A	999	1	999	68	WC015			1/17/2008 1:01	INR	0800584
0801159-01	FlashPoint 1020A	999	1	999	68	WC015			1/17/2008 1:01	INR	0800584
0801218-01	FlashPoint 1020A	999	1	999	68	WC015			1/17/2008 1:01	INR	0800584
0800584-BS1	FlashPoint 1020A	81	1	81	68		100		1/17/2008 1:01	INR	0800584
8011708-SCV1	FlashPoint 1020A	81	1	81			100		1/17/2008 1:01	INR	8011708
0800584-BLK1	FlashPoint 1020A	0	1	0	68	WC015			1/17/2008 1:01	INR	0800584

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STANDARD OPERATING PROCEDURE

Paint Filter Liquids Test

SW-846 Method 9095B

APPROVALS:

Area Supervisor: Heather L. Brady Date: 8-4-12
Heather L. Brady

QA Officer: Tom C. Boucher Date: 8-8-12
Tom C. Boucher

Laboratory President: Douglas E. Kriscunas Date: 8/20/12
Douglas E. Kriscunas

Procedure Number: GR-19-102
Revision Number: 1.2

Date Initiated: 1/28/97
Effective Date: 8/20/12

Date Revised: 8/3/12
Pages Revised: All

By: Heather L. Brady
Total Number of Pages: 8

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If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
<u>2-14-13</u>	<u>Heather L. Brady</u>	<u>2-14-14</u>

1.0 SCOPE AND APPLICATION

- 1.1 This procedure determines the presence of free liquids in a representative waste sample in compliance with the definition of "free liquid" found in 40 Code of Federal Regulations (CFR) 264.314 and 40 CFR 265.314.

2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 4th Edition, Final Update IV, Method 9095B, Paint Filter Liquids Test, Revision 2, November, 2001*

3.0 SUMMARY OF PROCEDURE

- 3.1 A representative amount of waste is placed in a 60 mesh paint filter.
- 3.2 If any portion of the material (liquid or otherwise) passes through and drops from the filter within the 5 minutes, the material is reported as containing free liquids.

4.0 PARAMETER OR COMPOUND LIST

- 4.1 Paint Filter Liquids Test

5.0 REFERENCED SOPs

- 5.1 TriMatrix SOP GR-15-102 *Laboratory Waste Disposal*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

- 6.1 The filter medium may separate from the filter cone if the waste is alkaline and has significant free liquid. This causes no problem if the sample is not disturbed and remains suspended in the filter cone.
- 6.2 Samples must be at room temperature to test. Do not test a frozen or hot sample.

7.0 SAFETY PRECAUTIONS

- 7.1 Wear a laboratory coat and approved safety glasses while in the 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.

- 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 exposure to the lowest possible level by adherence to established safety policies.
- 7.4.3 Material Safety Data Sheets are maintained on the laboratory inventory of all chemicals used in this procedure. Consult the MSDS for detailed chemical information.

7.5 Waste samples can be highly toxic and varied. Treat any exposure as potential danger and immediately decontaminate the exposure. Clean waste-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 Collect samples in inert plastic or glass bottles and stored at 0 - 6° C until the time of analysis.
- 8.2 A 100 g or 100 mL sample is required for the test. However, enough sample quantity must be collected to sufficiently represent the waste.
- 8.3 If it is not possible to obtain 100 mL or 100 g sufficiently representative of the waste, collect larger size samples in multiples of 100 g or 100 mL (200, 300, 400 g or mL). However, when larger samples are tested, divide into 100 g or 100 mL portions and test each separately. If any tested portion contains free liquids, the entire sample must be reported to have free liquids.
- 8.4 If a sample is measured volumetrically, it must lack major air spaces or voids.
- 8.5 Analysis must be performed within 28 days of sample collection.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

- 9.1 Conical paint filter, mesh number 60 ±5% (fine mesh size). Available at local paint stores such as Sherwin-Williams and/or Glidden.
- 9.2 Glass funnel
- 9.2.1 If the paint filter with waste cannot sustain itself on the ring stand, insert the filter in a fluted glass funnel or a smooth glass funnel with an aperture large enough to allow at least 1 inch of filter mesh to protrude.
- 9.2.2 The funnel must be fluted or have a large open aperture to support the paint filter yet not interfere with the movement of liquid through the filter mesh and into the graduated cylinder.

9.3 Ring stand and ring to hold the paint filter and/or the funnel

9.4 Graduated cylinder or beaker, 100 mL, to collect liquid

9.5 Timer or stop watch

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 There are no chemicals and/or reagents directly associated with this procedure.

12.0 STANDARDS PREPARATION

12.1 There is no standard preparation directly associated with this procedure.

13.0 SAMPLE PREPARATION

13.1 To assure uniformity and standardization of the test, cut sorbent pads, pillows and other materials which do not conform to the shape of the paint filter into small pieces and pour into the filter.

13.2 Accomplish sample size reduction by cutting sorbent materials with scissors, shears, a knife or other such device in such a way that preserves as much of the original sample integrity as possible by mixing sorbents enclosed in fabric with the resultant fabric pieces.

13.4 Reduce particle size of all samples to smaller than 1 cm. If necessary, check by passing through a 9.5 mm (0.375 inch) standard sieve. However, avoid grinding sorbent materials as grinding destroys sample integrity and may produce fine particles which would not normally be present. Such particles passing through the paint filter must be reported as "free liquid" based on the 40 CFR definition.

13.6 Lightly crush clay, silica gel, polymers and other brittle materials that do not conform to the filter to less than 1 cm particle size. Again, do not grind.

14.0 CALIBRATION PROCEDURES

14.1 There is no calibration directly associated with this procedure.

15.0 ANALYTICAL PROCEDURE

15.1 Assemble the test apparatus as shown in Attachment 21.1.

- 15.2 Place 100 g or 100 mL of representative sample in the paint filter. If necessary, use the funnel.
- 15.3 Let the sample drain for 5 minutes into the graduated cylinder.
- 15.4 If ANY portion of the test material collects in the graduated cylinder in the 5 minute period then the sample is reported to contain free liquids in accordance with 40 CFR 264.314 and 265.314.

16.0 CALCULATIONS AND DATA HANDLING

- 16.1 There are no calculations or data handling directly associated with this procedure.

17.0 DATA REPORTING AND DELIVERABLES

- 17.1 If any part of a tested sample (liquid or otherwise) or any part of one of several tested portions (liquid or otherwise) collects in the graduated cylinder, report that the result as "Fail" and narrate that the sample contains free liquids.
- 17.2 If no part of a tested sample (liquid or otherwise) or no part of one of several tested portions (liquid or otherwise) collects in the graduated cylinder, report that the result as "Pass" and narrate that the sample does not contain free liquids.

18.0 QUALITY ASSURANCE

- 18.1 A second sample aliquot must be tested as a quality control duplicate for each batch of up to 20 samples.
- 18.1.1 The duplicate test result must be the same as the original to be acceptable.
- 18.1.2 If not the same, review the sample tested for representative uniformity.
- 18.1.3 If both sample portions are representative, review the test performed for adherence to the written procedure.
- 18.1.4 If no problem is found with the procedure or sample uniformity, report the duplicated sample as containing free liquids and choose another sample at random from the batch to duplicate.
- 18.1.5 If more than one duplicated sample has discrepant results, the test is out of control and corrective action needs taken to locate the source of the problem.
- 18.2 Test 100 g of dry Ottawa sand as a negative paint filter liquids test. No sand must pass through the paint filter to be reported as not containing free liquids in accordance with the 40 CFR definitions.

19.0 ANALYST CERTIFICATION/METHOD VALIDATION

- 19.1 There is no quantitative demonstration of capability directly associated with this procedure.
- 19.2 Analyst testing for paint filter liquids must read and understand this procedure before conducting the test.
- 19.3 Signed documentation that the procedure has been read and understood is required to be on file with the quality assurance office.

20.0 REFERENCES

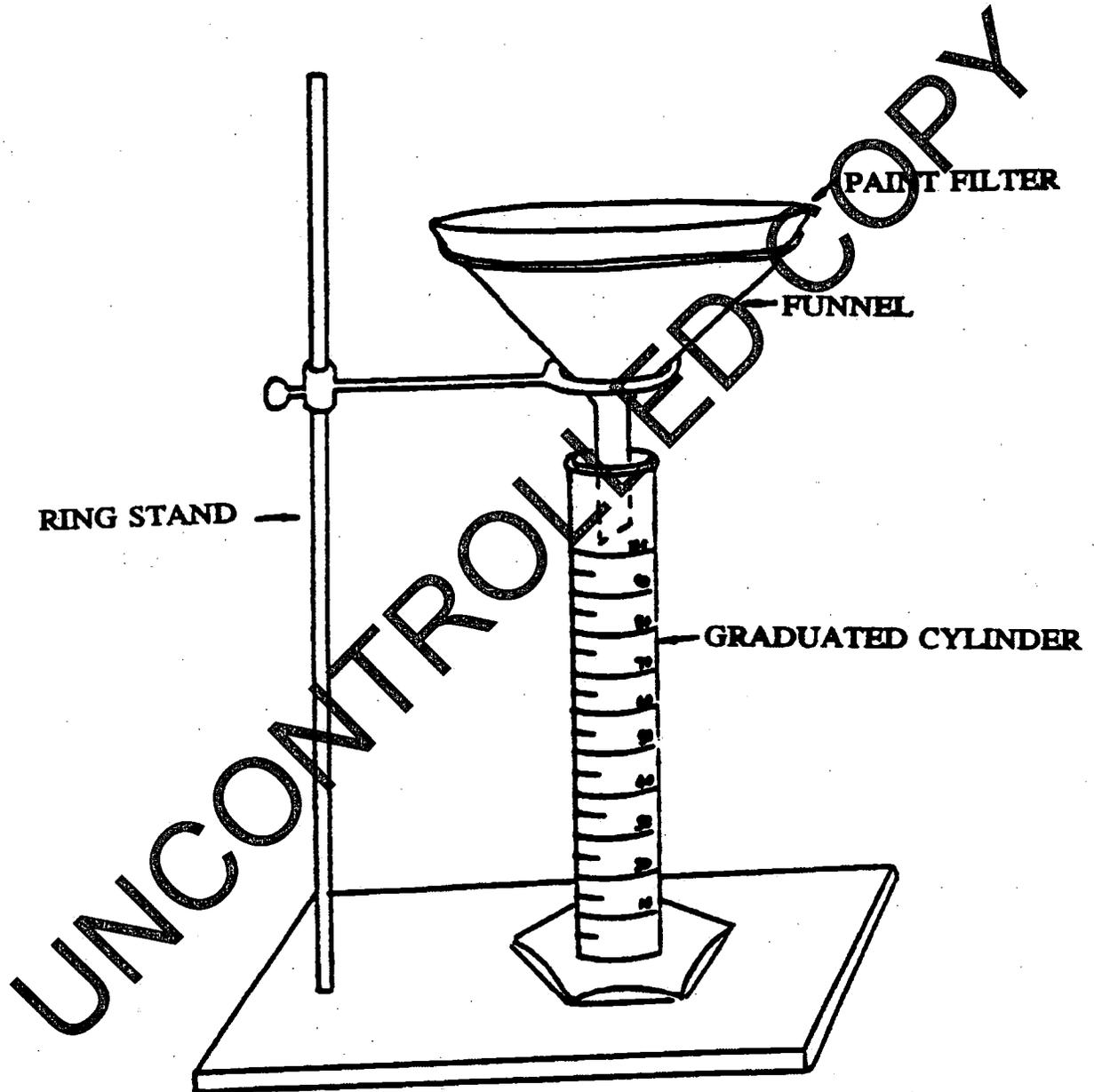
- 20.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 4th Edition, Final Update IV, Method 9095B, Paint Filter Liquids Test, Revision 3, November, 2004*

21.0 ATTACHMENTS/APPENDICES

- 21.1 Paint Filter Test Apparatus
- 21.2 Preparation Batch Report Example

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Attachment 21.1
Paint Filter Test Apparatus



SOP Name: Paint Filter Liquids Test
 SW-846 Method 9095B
 SOP Number: GR-19-102

page 8 of 8

Revision Number: 1.2
 Date Revised: 8/3/12
 Date Initiated: 1/28/97

**Attachment 21.2
 Preparation Batch Report Example**

TriMatrix Laboratories, Inc.

PREPARATION BATCH **0804851** Page 1 of 1

Printed: 5/16/2008 8:43:45AM

Inorganic - Wet Chemistry, Waste, General Inorganic Prep

Batch comments:
 (none)

Lab Number	Container	Prepared	By	Initial (g)	Final (mL) Client	Source ID	Spike ID	uL Spike	Comments
0804548-01	A	Apr-30-08 08:46	GEH	105.02	105.02					
<i>Paint Filter 9095</i>										
0804549-01	A	Apr-30-08 08:46	GEH	101.7	101.7					
<i>Paint Filter 9095</i>										
0804550-01	A	Apr-30-08 08:46	GEH	103.49	103.49					
<i>Paint Filter 9095</i>										
0804851-BLK1		Apr-30-08 08:46	GEH	100.06	100.06					Stawa Lab E#7090394
0804851-DUP1		Apr-30-08 08:46	GEH	103.98	103.98		0804550-01			

Comments:	Analyst Initials:
-----------	-------------------

bch_TM_byAnalysis.rpt

APPENDIX D
SUBCONTRACTOR STANDARD OPERATING PROCEDURES

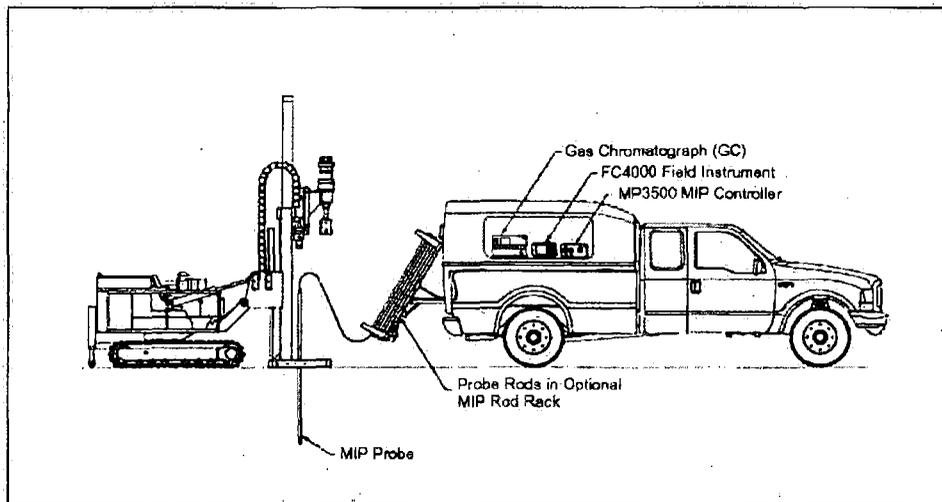
GEOPROBE® MEMBRANE INTERFACE PROBE (MIP)

Standard Operating Procedure

Technical Bulletin No. MK3010

PREPARED: May, 2003

REVISED: April, 2012



**THE MIP SYSTEM MAY BE DEDICATED TO A SINGLE CARRIER VEHICLE
FOR USE IN TANDEM WITH MULTIPLE GEOPROBE® MACHINE MODELS**



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1.0 OBJECTIVE

This document serves as the standard operating procedure for use of the Geoprobe Systems® Membrane Interface Probe (MIP) used to detect volatile organic compounds (VOCs) at depth in the subsurface.

2.0 BACKGROUND

2.1 Definitions

Geoprobe®: A brand name of high-quality, hydraulically-powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and testing, soil conductivity and contaminant logging, grouting, and materials injection.

**Geoprobe® is a registered trademark of Kejr, Inc., Salina, Kansas.*

Membrane Interface Probe (MIP): A system manufactured by Geoprobe Systems® for the detection and measurement of volatile organic compounds (VOCs) in the subsurface. A heated probe carrying a permeable membrane is advanced to depth in the soil. VOCs in the subsurface cross the membrane, enter into a carrier gas stream, and are swept to gas phase detectors at ground surface for measurement.

2.2 Discussion

The MIP is an interface between contaminants in the soil and the detectors at ground surface. It is a mapping tool used to find the depth at which the contamination is located, but is not used to determine concentration of the compound. Two advantages of using the MIP are that it detects contamination in situ and can be used in all types of soil conditions.

The MIP is a logging tool used to make continuous measurements of VOCs in soil. Volatile compounds outside the probe diffuse across a membrane and are swept from the probe to a gas phase detector at ground surface. A log is made of detector response versus probe depth. In order to speed diffusion, the probe membrane is heated to approximately 121°C. (Refer to Figure 2.1).

Along with the detection of VOCs in the soil, the MIP also measures the electrical conductivity of the soil to give a probable lithology of the subsurface. This is accomplished by using a dipole measurement arrangement at the end of the MIP probe so that both conductivity and detector readings may be taken simultaneously. A simultaneous log of soil electrical conductivity is recorded with the detector response.

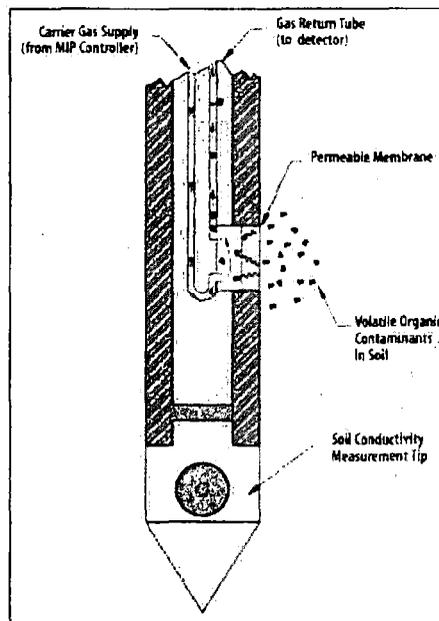


Figure 2.1: Diffusion across the membrane.

Interpretation of electrical conductivity (EC) logs comes with field experience. It is very important that soil core samples are taken to confirm lithologic changes as each EC log is unique per site. As a generalization, a high conductivity reading indicates a smaller grain size and a low conductivity reading indicates a larger grain size (See Fig. 2.2).

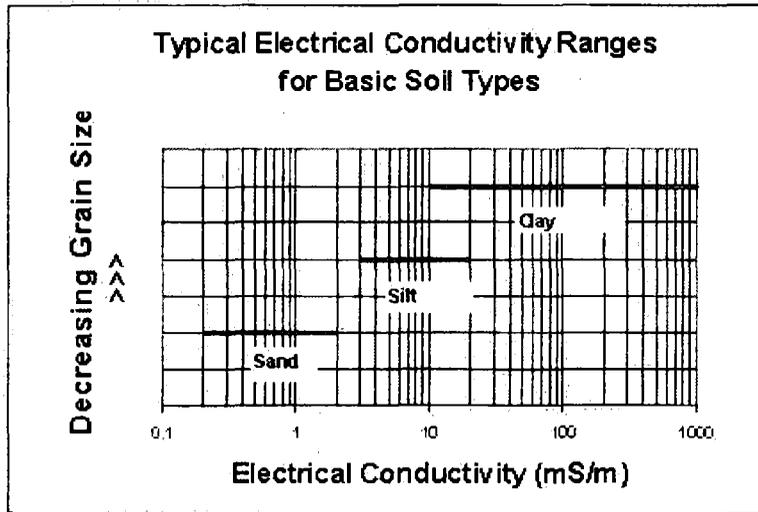


Figure 2.2: Generalized Electrical Conductivity Readings.

3.0 MIP/EC Interferences

- 3.1 Detector saturation may require a short period of time for the detector to return to baseline after a log has been performed in higher concentrations. The MIP system can be used in free product environments with the operator monitoring and making the necessary adjustments to the detector and software gain/attenuation settings to account for the higher voltage readouts.
- 3.2 The MIP system can be operated in a wide range of contaminant concentrations from low dissolved phase to free phase materials. During a log and the removal of the tool string, contaminants can absorb onto the surface of the membrane and trunkline material causing elevated detector baseline signals. It is very important that the probe and trunkline system is clean enough to see the low concentrations typically used in the chemical response test. Not adequately decontaminating the probe prior to performing a response test can elevate the concentration of the standard causing an inaccurate high response to the specific concentration of standard that was prepared for the test.
- 3.3 Electrical conductivity can provide false positives or higher than expected readings when the soil is impacted by ionic plumes (chloride, nitrate) originating from, but not limited to: agriculture practices, seawater, salt storage, mining practices. Encountering metallic objects in the subsurface can also result in high EC readings.
- 3.4 Some silt and clay soils will not have the typical ionic composition that an operator may be used to for similar soils. This can result in lower than expected readings and perhaps cause misidentification of the associated soil zone based on typical response of a coarser grain material. This can occasionally be found in clays that have had the minerals leached out or in intermixed silt-sand zones.

4.0 Tools and Equipment

The following equipment is needed to perform and record MIP logs. Basic MIP system components are listed in this section in section 4.1 with optional equipment listed in section 4.2. Refer to Appendix V for a detailed illustration of the GC1000 setup configuration. Appendix VI shows the common MIP probe tool string diagrams. There may be more required tools as determined by your specific model of Geoprobe® direct push machine.

4.1 Basic MIP System Components

Description	Quantity	Part Number
Field Instrument	(1)	FC5000 / FI6000
MIP Controller	(1)	MP6500 / MP6505
Gas Chromatograph with PID, FID and XSD	(1)	GC1000
DI Acquisition Software	(1)	MP3517
MIP Probe	(3)	MP6520/MP8520
MIP PEEK Trunkline, 150-ft (45 m) length	(2)	14929
MIP Connection Tube	(3)	31641
MIP Adapter and Drive Head	(3)	20712
Agilent ADM 1000 Digital Flow Meter	(1)	17463
Hydrogen Gas Regulator	(1)	10344
Nitrogen Gas Regulator	(1)	13940
Vertical Gas Bottle Rack	(1)	ML2500
Stringpot (linear position transducer)	(1)	SC160-100
Stringpot Cordset	(1)	SC161
Stringpot Mounting Bracket (6600/7700)	(1)	16791
Stringpot Foot Bracket (6600/7700)	(1)	11751
Stringpot Piston Weight	(1)	SC112
Slotted 1.5" Drive Cap	(2)	13722
MIP Service Kit	(1)	MP6515
Drive Cushion (GH60)*	(1)	23321
Rod Wiper, 1.25/1.5" Rods	(1)	23852
Rod Wiper Weldment	(1)	23633

4.2 Optional Accessories

Description	Quantity	Part Number
Heated Trunkline Control Box	(1)	MP7000
Heated Trunkline, 100-ft (30m) length	(1)	MP7100
Heated Trunkline, 150-ft (46m) length	(1)	MP7150
Heated Transfer Line, 8-ft (2.4m) length	(1)	MP7010
Roll-out Rod Rack (30-1.5in rods)	(1)	20400-30
Rod Grip Pull Handle, for GH40 hammer	(1)	GH1255
Rod Grip Pull Handle, for GH60 hammer	(1)	9641
Stringpot Mounting Bracket (7822)	(1)	41932
Stringpot Foot Bracket (7822)	(1)	41993
Water Transport System	(1)	19011

*For Geoprobe® 66- and 78-Series Direct Push Machines only.

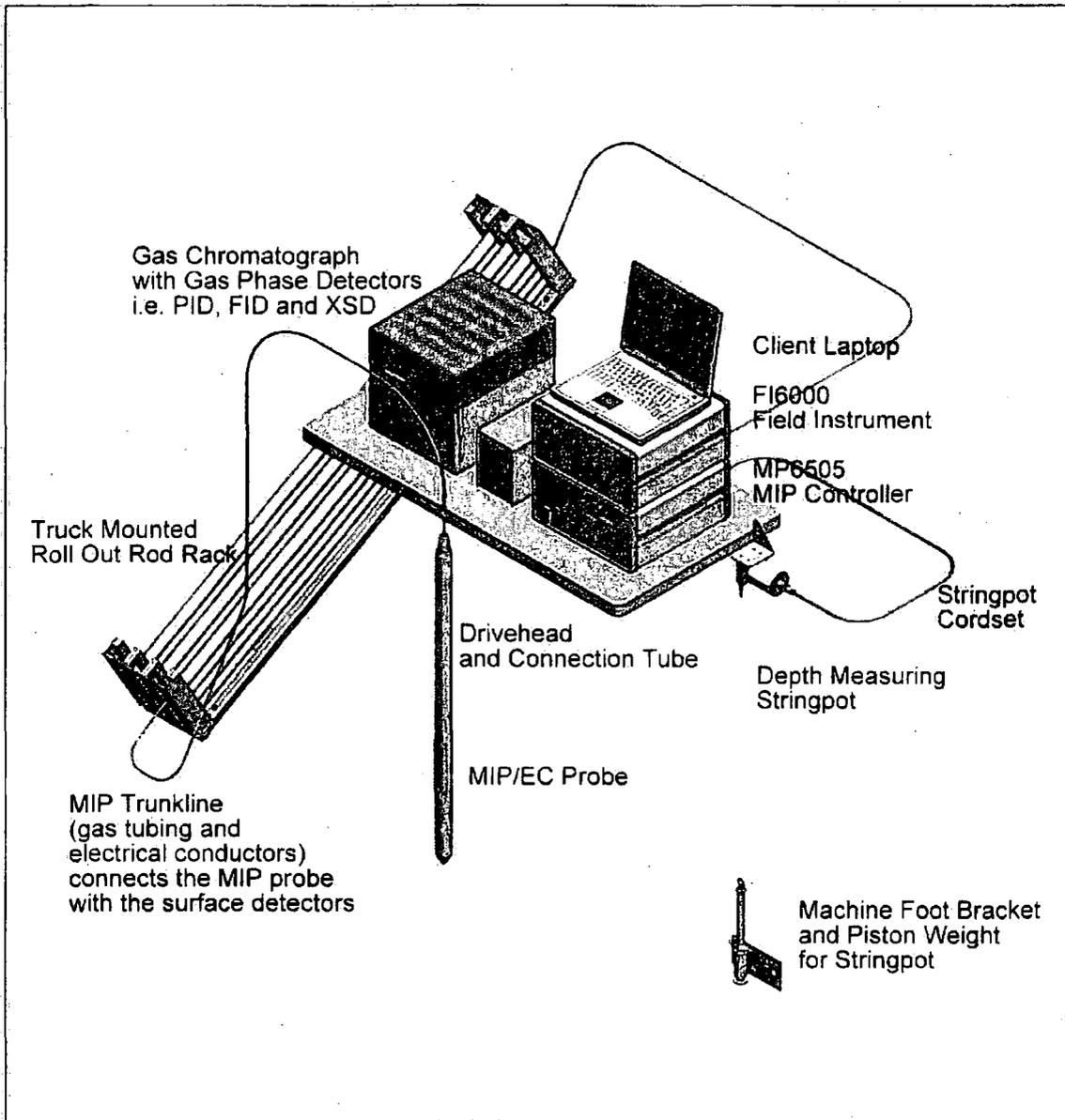


Figure 4.1: MIP System Components

5.0 Quality Assurance/Quality Control

Quality assurance (QA) is performed before and after each log to validate that the equipment being used is capable of generating good data. With MIP, chemical response tests (Fig. 5.1) must be performed to ensure that the probe, trunkline and series of detectors are working properly. The electrical conductivity (EC) portion of the MIP system is tested using an EC dipole test jig (Fig. 5.4)

Quality control (QC) is performed during and after each log is generated. Log QC will answer the following questions to ensure that the data is good and makes sense:

1. Does the log look correct? Does the electrical conductivity appear to be in an acceptable range? Is there anything seen in the log that would make you suspect that the system wasn't working correctly, i.e. a loss of temperature or gas pressure of the system.
2. Response Consistency? As more logs are completed do they show general consistency of EC and contaminant response? Review a cross section of logs in the DI-Viewer (Appendix VI).
3. Repeatability? Replicate logs may be run every 10 to 20 locations to verify repeatability.
4. Are my lithogy changes consistent with physical soil cores? Take continuous or discrete confirmation soil samples to confirm your lithogy changes in EC.
5. Do my detector responses make sense for contaminant concentration. This must be verified by the collection of water or soil samples for lab analysis to confirm contaminants and their concentrations.

5.1 Chemical Response Test:

Response testing is an important quality assurance measure used to validate each log by proving that the integrity of the detector system is intact. Without running a response test, the operator will have no idea if the detector system is operating consistently or potentially even at all. Detector response heights should be monitored and can be graphed to evaluate membrane performance. With increased membrane footage, detector response will fall off indicating that it is time to change the membrane (see Appendix III). Response testing also enables the operator to measure the chemical trip time. This is the time it takes for the contaminant to travel through the trunkline from the probe to the detectors. This time needs to be entered into the MIP software to accurately plot the contaminants depth position.

5.1.1 Preparation of the Stock Standard

The following items are required for preparing the stock standard:

- Neat sample of the analyte of interest (i.e.: Benzene, Toluene, TCE, PCE, etc.) purchased from a chemical vendor
- Microliter syringes (recommended to have: 500 and/or 1,000 μ L syringes).
- 25-mL or 50-mL Graduated cylinder
- Several 40-mL VOC vials with labels
- 25mL Methanol

Preparation of the stock standard is critical to the final outcome of the concentration to be placed into the testing cylinder.

1. The total volume of methanol and the compound added should equal 25mL.
2. Pour methanol into graduated cylinder to the 23.5-24mL mark, the volume depends upon the compound density (Table 5.1).
3. Pour the methanol from the graduated cylinder into a 40-mL VOC vial.
4. Add the appropriate volume of desired neat analyte into 40-mL VOC vial containing methanol. The required volume of neat analyte for seven common compounds is listed in Column 3 of Table 5.1. The equation at the bottom of this section shows how to calculate the appropriate neat analyte volume for other compounds of interest given the appropriate density.

5. Label the vial with the name of the standard (i.e. Benzene, Toluene, TCE, PCE), concentration (50mg/mL), date created, and created by (your name). This is the Stock Standard.
6. If stock standards are kept cold in a refrigerator they can last up to one month otherwise they should be made up more frequently as often as every 3 days if there is not cooling during the summer months. The more volatile the compound the quicker it will lose its concentration.

Table 5.1
Density and required volumes of neat compounds used to make a
50mg/mL stock standard into 25 ml of methanol.

Compound	Density (mg/ μ L)	Volume of Neat Standard Required to prepare Working Standard (0.5 L)
Benzene	0.876	1426
Toluene	0.867	1442
Xylenes	0.860	1453
Methylene Chloride	1.335	936
Carbon Tetrachloride	1.594	784
Chloroform	1.480	845
Trichloroethylene	1.464	854
Perchloroethylene	1.623	770

25mL (methanol) x 50mg/mL = 1250mg
 1250mg x 1/density of analyte = amount of neat material to be placed with methanol to make up 25mL total volume

Example: Preparation of 50 mg/mL Benzene standard.

$1250 \text{ mg} \times 1/0.8765 \text{ mg}/\mu\text{L} = 1426 \mu\text{L}$
 Use 1426 μ L of neat Benzene in 23.5mL of Methanol to get a 50 mg/mL stock standard.

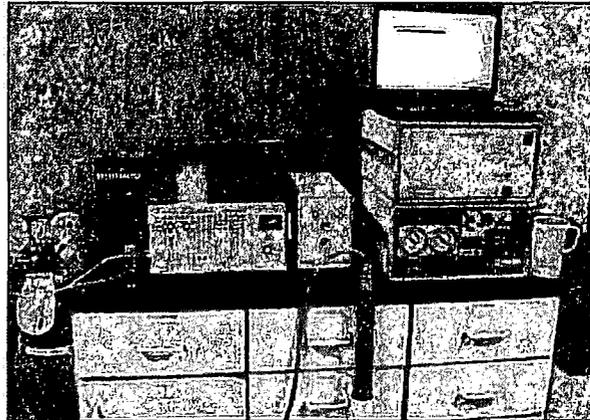


Figure 5.1: The MIP probe is placed into a steel or PVC pipe containing the standard solution.

5.1.2 Preparation of the Working Standard and Performing the Response Test

The following items are required to perform response testing:

- Microliter syringes (recommended to have: 10, 25, 100 & 500 μ L syringes).
 - Testing cylinder made from a nominal 2-in. PVC pipe with a length of 24 in.
 - 0.5 L plastic beaker or pitcher
 - Supply of fresh water, 0.5 L needed per test
 - Stopwatch
1. On the FI6000 and the DI-Acquisition software you will begin a new log and proceed to the response test screen. The detector signals should be stable before proceeding. On the FC4000 and FC5000 system, access the MIP Time software and view the detector vs. time data.
 2. Measure out 500mL of tap or distilled water and place into the testing cylinder.
 3. Using Table 5.2, determine the desired volume of stock standard to place into the 500ml measured volume of water. This is the Working Standard.
 4. Pour the working standard into a nominal 2-inch x 24-inch PVC pipe and immediately insert the MIP into the solution (Fig. 5.1). Leave the probe in the test solution for 45 seconds. At the end of 45 seconds, place the probe back in into a clean water source.
 5. The chemical response trip time can be determined from the results on the Pre-Log Response Test. Using Fig. 5.2 the trip time would be approximately 55 seconds. Additional typical response test graphs are located in Appendix I.
 6. A new, fresh working standard needs to be made for each test, it cannot be reused.

Table 5.2
Volume of stock standard and final concentration when making working standards.

Volume of Stock 50mg/mL Standard (μ L)	Final Concentration (mg/L or ppm) in 0.5L
10	1.0
100	10
1000	100

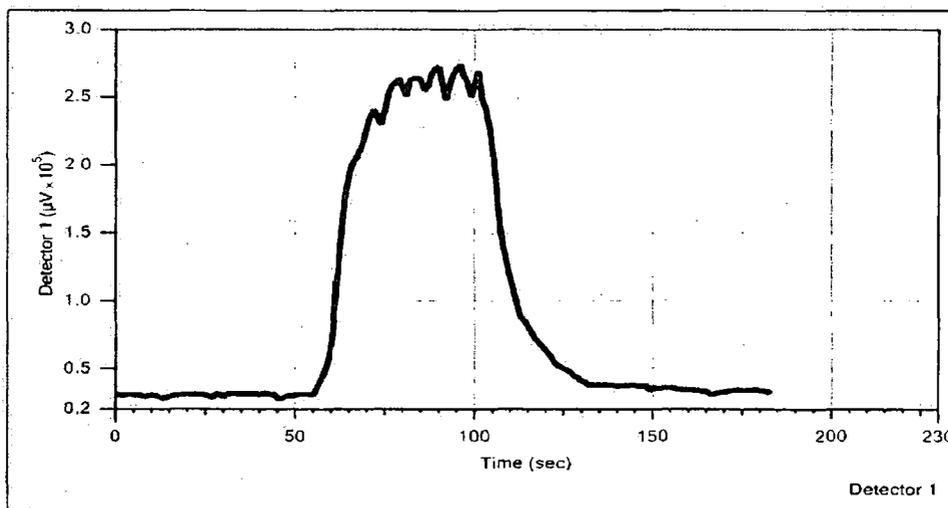


Figure 5.2: SRI PID Response Test - 10 ppm Benzene.

5.2 Operation of EC Dipole Test Jig

On the FI6000 and the DI-Acquisition software the EC dipole test screen will open up after the chemical response test is completed. When ready place the low (brass) side of the EC Dipole test jig (Fig 5.3) between the EC dipole and body of the probe and start the low level test, hold for 5 sec until the system captures the data (Fig 5.4). Repeat for the high (stainless steel) EC test. These tests should result in readings of 55mS/m and 290mS/m \pm 10%.

In the FC4000 and FC5000 acquisition system these readings will need to be taken in the logging screen just prior to the beginning of the log.

If the EC readings do not pass, the DI Acquisition (FI6000) software will prompt the user to proceed through a series of troubleshooting tests (these tests used to be the standard test for EC in the FC4000 and FC5000 software). These tests will check the calibration of the EC board as well as the continuity and isolation of all of the wires in the system to determine the reason EC Tests loads have failed. This will give the operator an idea what needs to be done to fix the problem.



Figure 5.3: EC Dipole Test Jig.

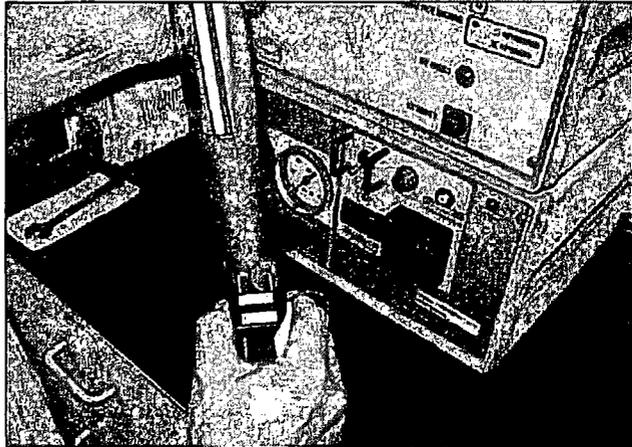


Figure 5.4: Operation of the EC Dipole Test Jig.

6.0 Recommended Minimum MIP Response Test Levels and Maintenance Tips

Geoprobe Systems recommends the following guidelines as minimum MIP response test values for performing MIP logging.

Detector systems can vary in the level of response for a given chemical concentration depending on detector age, model, and maintenance performed. However, it should be expected that a detector system would be able to provide at least the following level of response in a chemical response test:

<u>Chemical & Concentration</u>	<u>Detector Response</u>	<u>Baseline Noise</u>
10ppm Benzene (see table 5.2)	PID-10,000 μ V	<3,000 μ V
10ppm Trichloroethylene (see table 5.2)	PID-5,000 μ V, XSD 10,000 μ V	<3,000 μ V

If these minimum response test levels are not achievable or throughout the day or project the detector sensitivity falls below these levels, the operator should perform maintenance on the system to enhance the sensitivity of the detectors. Corrective actions could include:

- Changing MIP membrane (see section 9.0)
- Making a fresh chemical stock standard (see section 5.1.1). It does not take long for a volatile chemical standard to lose the original concentration.
- Cleaning the PID bulb
- Replacing the PID bulb
- Checking and adjusting detector gas flows - especially in the FID.
- Replacing the XSD probe assembly
- Replacing the XSD reactor core
- Decreasing trunkline carrier gas flow
- Replacing the trunkline (an old trunkline can be a source of contaminant phase buildup. This will reduce detector sensitivity by causing contaminant dispersion in the trunkline which results in reduced response levels as well as delayed trip times).

7.0 Field Operation

1. Power on the generator.
2. Open the gas cylinders that will be used for the MIP system (i.e. nitrogen, hydrogen, air, etc.).
3. Power on the GC and detectors and allow them to warm up (min. 20 minutes) to set temperature.
4. Check the carrier flows of the system and psi on the mass flow controller. Compare these numbers to previous work.
5. Power on the MIP Controller, Field Computer or the Field Instrument and laptop computer.
6. Start the Acquisition software and start a new log.
7. Perform the chemical response test (Section 5.1.2) and record the height of the peak response and the trip time into a field notebook. Refer to Figure 5.2 and Appendix III.
8. Record the system parameters in a field notebook at this time (i.e. flow, pressure, trip time, detector baseline voltages).
9. Complete the EC Dipole test (Section 5.3) and finish setting up the log.
10. Connect the stringpot cable to the stringpot and the stringpot wire to the weight located on the probe foot and pull keeper pin so the weight will drop to the ground.

NOTE: Do not allow the stringpot cable to snap back into the stringpot housing at a high rate of speed. This will ultimately damage the stringpot transducer.

11. Place the drive cushion onto the probing machine head.
12. Place a slotted drive cap to the MIP drive head.
13. Place the rod wiper donut on the ground and insert the point of the MIP probe into rod wiper opening.
14. Align the probe exactly straight and advance the probe to the starting depth: MIP membrane even with the ground surface.
15. Place the trigger switch in the "ON" position.
16. Advance the probe at a rate of 1ft/min meaning: advance 1 ft (30 cm) in 15 seconds and then hold at depth for 45 seconds, then advance to the next depth interval (1 foot) over 15 seconds and wait for 45 seconds. Do this until the predetermined log depth or until refusal is attained.

NOTE: If there is a loss in MIP pressure or temperature during the logging process, stop and evaluate the problem using the troubleshooting guide located in Appendix II.

NOTE: Refusal is attained when it takes longer than 1 minutes of continuous hammering to advance the probe one foot. This is the maximum time to reach one foot of probe travel.

17. When the MIP log is complete, turn the trigger off and slowly return the stringpot cable into the stringpot housing.
18. Turn off the heater switch to the probe during tool string retraction so no as few contaminants as possible are diffused through the membrane and into the trunkline during retraction.
19. Raise the probe foot of the direct push machines foot assembly and place the rod wiper holder under it to keep it in place during rod retraction.
20. Pull the probe rod string using either the Geoprobe[®] rod grip pull system or a slotted pull cap.
21. When the MIP probe reaches the surface, clean the probe and membrane well with a detergent/water mix and rinse off well.
22. Now turn the probe heat back on to back off the membrane. Make sure the probe membrane and trunkline are clean of contaminants and the detector baselines are stable prior to running a post log response test. View the detector activity in the response test screen.
23. When the baselines are stable run a post log response test. These response test results should be written down in the field notes and compared to the initial test. This system check ensures the data for that log is valid.
24. When using the FI6000, the data will be saved into your designated folder on your laptop in a compact .zip file. If you are using a FC5000 the data is saved on the field computer and the inserted flash card. When the log is complete the log files and response test files will need to be transferred to a field laptop for viewing on the DI Viewer.
25. Data from the MIP log can now be graphed and printed using the DI-Viewer software (Appendix IV).

8.0 GC Signal Adjustments

8.1 Dilution/Attenuation Changes

GC systems vary in signal output ranges such as 0-1V (typical for HP GCs) and 0-5V (typical for SRI & Shimadzu GCs) which means that when detector signals go beyond this voltage in the output, the acquisition software will display a flat line at the maximum voltage of 1 or 5Volts unless this signal output has been rescaled.

In highly contaminated soil regions (e.g. free product) detectors may flat line or reach a maximum signal output before they reach the observed signal of the contaminant in contact with the membrane. For example, on SRI GCs to be able to observe the actual response beyond the maximum output signal in these high response areas the PID gain switch should be adjusted from high to medium and the software attenuation set to 10. What this does is adjusts the detector output signal down by a factor of 10 which must then be readjust back up by the same factor of 10 in the acquisition software. To accurately map the grossly contaminated zones rescaling of the detectors must be done.

Detectors operated through a HP5890 GC have a 1V maximum signal output and the attenuation settings are based on a 2^x multiplication scale $x = \text{HP GC Range}$ and the corresponding attenuation in the MIP software. SRI and Shimadzu GCs have maximum signal outputs of 5V and the attenuation settings are based on a 10^x multiplication factor.

Table 8.1
Gain/Attenuation Settings on the GC detectors and the Acquisition software.

HP GC* Range	FI6000 Attenuation		SRI GC Gain	XSD Gain	FI6000 Attenuation
0	1		High	High	1
1	2		Medium	Medium	10
2	4		Low	Low	100
3	8				

*- The detectors on the HP GC can have attenuation settings up to a range of 7 on the GC corresponding to an acquisition software multiplication value of 128.

9.0 Replacing a Membrane on the MIP Probe

A probe membrane is considered in good working condition as long as two requirements are met:

1. Adequate signal response is achieved during the chemical response tests to see the required detection limits.
2. The difference between the supply and return flow has not increased by more than 3mL/min from the original settings. (A digital or bubble flow meter should be kept with the system at all times).

If either one of these requirements are not met, a new membrane must be installed as follows.

1. Turn the heater off and allow the block to cool to less than 50° C on the control panel readout.
2. Clean the entire heating block with water and a clean rag to remove any debris.
3. Dry the block completely before proceeding.
4. Remove the membrane using the membrane wrench (Fig. 8.1). Keep the wrench parallel to the probe while removing the membrane to ensure proper engagement with socket head cap screw.

NOTE: Do not leave the membrane cavity open for extended periods. Debris can become lodged in the gas openings in the plug.

5. Remove and discard the copper washer as shown in Figure 8.2. Each new membrane is accompanied by a new copper washer. **Do not reuse the copper washer.**
6. Clean the inside of the membrane socket with a q-tip and methanol removing dirt and debris that will be present.
7. Insert the new copper washer around the brass plug making sure that it sits flat on the surface of the block.
8. Install the new membrane by threading it into the socket. Use the membrane wrench to tighten the membrane to a snug fit. Do not over-tighten.
9. Turn the gas on and leave the heater off. Apply water to the membrane and surrounding area to check for leaks. If a leak is detected (bubbles are formed in the water), use the membrane wrench to further tighten the membrane.
10. Use a flow meter to check carrier flow. The difference between the supply flow from the MP6505 and the return flow from the trunkline should be less than 3ml/min. Record the values in a field notebook.

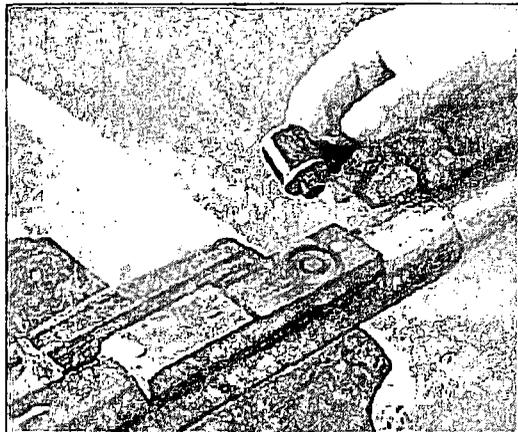


Figure 8.1: Unthread the membrane from the probe block.

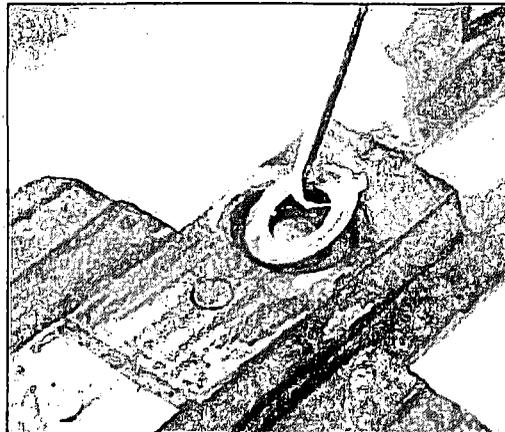


Figure 8.2: Remove and discard the copper washer.

APPENDIX I

Typical Response Test Data

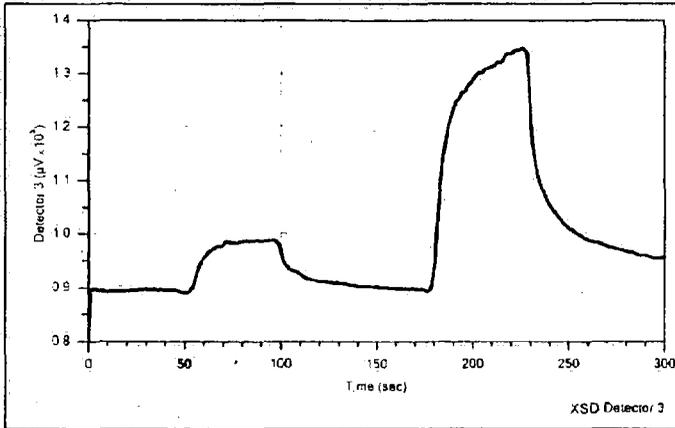


Figure 1: Chemical Response Test: TCE 1 & 5ppm on XSD.

System Parameters:

MP6520 Probe with 121°C setpoint
150' PEEK Trunkline
40ml/min of Nitrogen Carrier Gas
XSD Temperature of 1,100°C

System Response:

1ppm – 9,000μV
5ppm- 45,000μV

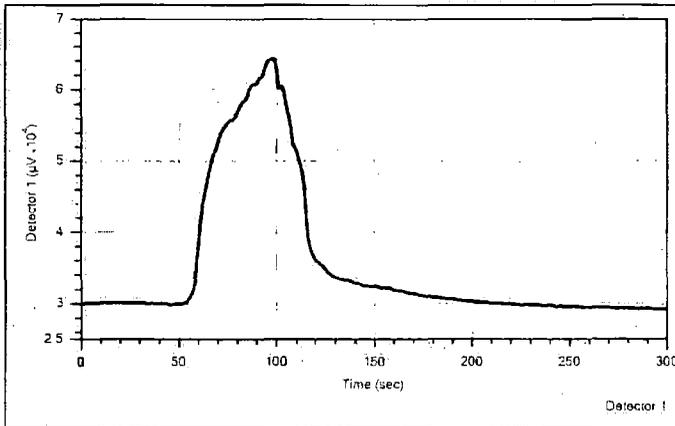


Figure 2: Chemical Response Test: Benzene 5ppm on PID.

System Parameters:

MP6520 Probe with 121°C setpoint
150' PEEK Trunkline
40ml/min of Nitrogen Carrier Gas
PID Lamp intensity

System Response:

5ppm- 35,000μV

APPENDIX II

Troubleshooting Guide

Loss of Pressure 1-2 PSI

- Punctured membrane: Are there any obvious holes in the membrane with bubbles streaming out of them? Replace membrane.
- Membrane leaking out of the face – heavy frothing of bubbles on membrane face but no obvious punctures in membrane. **With the heat off**, place your thumb over the membrane, if the pressure goes back up to the gas pressure prior to the boring the pressure and flow loss is due to a leak in at the membrane face. Replace the membrane.
- Swagelok fitting connecting one of the trunkline gas lines to stainless steel gas line of the probe is loose. Check with soapy water, if bubbles build, fix by slowly tighten the gas line 1/16" nut to the probe.
- Examine for cuts, kinks & cracks in the length of the observable gas line. Expect to see bubbling when MEOH or soapy water is placed on it. Cut gas line prior to this and replace nut and ferrule and reconnect onto the probes steel gas line connection.
- Broken gas line somewhere else up the trunkline. Confirmed when trunkline connections are removed from the probe and close coupled. The carrier gas supply and return should be within 2ml/min, if it is >5ml/min first check with soapy water at the connecting nuts and exposed gas line then look for cuts in throughout the trunkline and see if they will show bubbles with soapy water placed on them. If this is seen you will likely need to change the trunkline.

Loss of Pressure >5 PSI

- Large puncture in membrane. Either visible puncture or observable streaming bubble when soapy water or methanol placed on membrane. Replace membrane.
- Loosen the 1/8" Swagelok nut on gas line. Check and carefully tighten.
- Broken gas line in the probe. Compare the supply versus return flow values (should ≤ 2 ml/min) of trunkline connected with the probe and with a close coupled trunkline. If close coupled supply/return flow is good but connected to the probe shows a big leak, there is a break in the probe. This may be seen with soapy water placed on the edges of the heater block or on the top of the probe where the connections come out. If this produces bubbles it confirms a broken internal line or connection point. Replace the probe.

Flash Warning:

The DI acquisition system, operated with the FI6000 field instrument, will flash a large warning screen – MIP pressure out of Range - to the operator if the probe pressure (PSI) changes over 1 PSI from the initial starting MIP pressure of the log. This alerts the operator that something in the system has changed and the operator can take the necessary precautions for a punctured membrane, broken gasline or a plug in the system.

Increase in Pressure (clearing a blockage)

After setting the mass flow, an increase of more than 3 PSI over the original set pressure indicates a potential blockage, especially if you can verify that the pressure first dropped a 2-5 PSI prior to rising toward 20PSI.

- Shut off the Nitrogen carrier gas flow ASAP. Do this by turning off the black regulator knob on the MIP controller or removing the carrier gas supply line from the breakout panel or the back of the MIP controller.
- Remove the tools from the ground.
- Look for a hole in the membrane and water or dirt got into the up-hole gas line just behind the membrane.
- Remove connection tube and membrane.
- Remove the trunkline gas lines from the top of the probe. Take note of which one had the gas flow coming out because this is the line that will be plugged.
- Look for any obvious particles in either holes behind the membrane or in the gas line at the top of the probe. If any are evident attempt to remove them.
- Take the return gas line at the surface and connect it to the supply gas connection on the breakout panel or on the back of the MIP controller.
- Place the probe end of this line into a jar of methanol to see if the line is clear which is evident by streaming bubbles. If there are no bubbles, increase the flow to try to expel the blockage. If this does not work you may need to cut back the trunkline.
- To clear out the probe take a 5 ml plastic syringe (or a 3 foot section of Teflon/PEEK gas line will work) filled with methanol and attempt to inject through the plugged gas line at the top of the probe. If it clears it will shoot the methanol in an arcing stream out one of the ports in the plug that sits behind the membrane.
- The probe must be dried of the methanol which can be accelerated by heating the probe. Don't reconnect the trunkline to the detectors until you are sure the blockage is clear and the methanol is out of the system.
- If the blockage cannot be cleared a new probe or trunkline will have to be connected.

Blinking Temperature Light

If the temperature light on the MP6505 begins blinking in an unreadable number, it means that there is an open thermocouple in the system.

- To complete the log in progress, replace the thermocouple for the trunkline with a thermocouple wire and twist-tie the wires together. This will fool the system to thinking there is continuity of the thermocouple wire and allow you to finish a log. The probe will continually heat set up this way and if left on when out of the ground it will overheat. When the log is complete remove the tricked thermocouple and remove tools from the ground.

When you have the probe out of the ground, replace the thermocouple as follows.

- Remove the connection tube from the probe.
- Check the crimp connections of the thermocouple wires from the trunkline to the probe.
 - If one of the crimp connections has broken then strip back the wire on both sides of the thermocouple – probe and trunkline ends and reconnect in a new crimp connection and see if the probe temperature comes back.
 - If the thermocouple connection is good, the thermocouple wire in the probe has likely broken. Cut off the crimp connections of the thermocouple wires between the probe and the trunkline. Check the resistance between the red and yellow thermocouple wires coming out of the probe. A resistance reading of approximately 40ohms indicates that the thermocouple is good reconnect. If they are open (O.L.) or mega ohms then the leads are broken on the thermocouple. Replace the thermocouple.

To check the trunkline thermocouple wires, measure each wire from top to bottom. The resistances will be different between the two colored wires but should be somewhere approximately 50 ohms – 150ohms for the length of the trunkline. The resistances will also increase with an increase in trunkline length.

- If they are open (no resistance) then there is a break in the trunkline. Replace the trunkline.

Spiking the Pressure and/or Temperature Data

If spikes show up in the temperature or pressure data especially when related to hammer strikes it is likely an intermittent break in the thermocouple connection. Spiking of the temperature may reach single point readings of 250°C in the data but may not be visible when watching the temperature display on the MIP controller.

- When you check the resistance between the two thermocouple wires they may check out at approximately 40 ohms, however there likely is an intermittent break in the wire.
- Replacing the thermocouple should eliminate the pressure and temperature data spikes.

Probe Not Reaching Temperature

If the heater light is on but the temperature seems low (<100°C with a set point of 120°C) a heater may have broken in the probe.

- Check the resistance of the heater wires.
 - If a heater is broken the resistance will be over 40 ohms. The probe needs to be replaced.
 - Two good heaters will read approximately 22 ohms on the MP6520, MP8520 and MK6530.
- Check to see if the thermocouple has pulled a few inches out of the probe.
 - If the thermocouple duct has broken and pulled back away from the probe, the probe will need to be replaced and rebuilt.
 - A thermocouple can unscrew and vibrate loose out of the thermocouple duct connection if it is not secured with shrink tubing or electrical tape. Reseat back into the leur-lock connection and secure. When the thermocouple pulls away from the probe it measures the probe temperature in the wrong location.

Flash Warning:

The DI acquisition system, operated with the FI6000 field instrument, will flash a large warning screen – Temperature out of Range - to the operator if the temperature goes outside of a set range from the setpoint temperature of 121°C. This alerts the operator that something in the system has failed and the operator can take the necessary precautions for a broken probe heater or thermocouple problem.

System Explanations and Warnings

- **MIP Flow**

MIP flow is the carrier gas flow set by the MIP controller. This flow is supplying carrier gas to the trunkline and probe and is typically set to approximately 42ml/min. This parameter may be monitored by the DI-Acquisition system if the operator has the necessary components in their MIP Controller. The return flow, or Flow-R, is the flow coming back to the GC up the return gas line. Flow-S and Flow-R should be within 3-4ml/min and are usually much closer.

- **MIP Pressure**

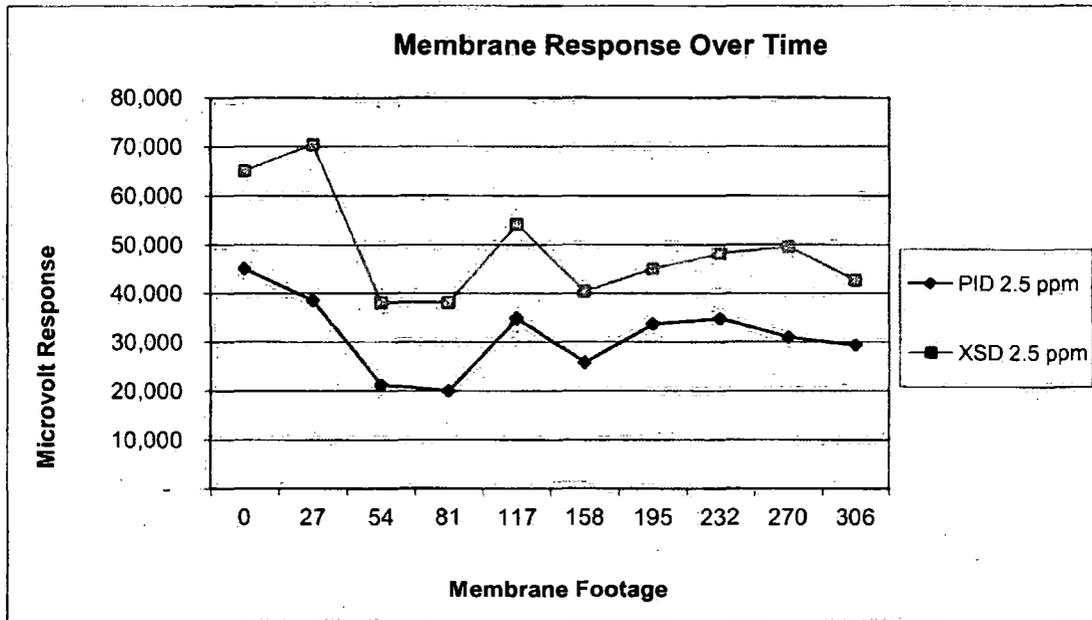
The MIP pressure is the back pressure of the carrier gas as it moves through the trunkline and probe. This is monitored digitally on the DI-Acquisition screen as well as by an analog pressure gauge on the front of the MIP controller. The MIP pressure is directly related to the MIP return flow (Flow-R). If the MIP pressure falls, the return flow has also dropped, if the MIP flow (Flow-S) has remained the same then there is likely a punctured membrane or problem with the gas lines.

APPENDIX III

Membrane Performance Control Charts

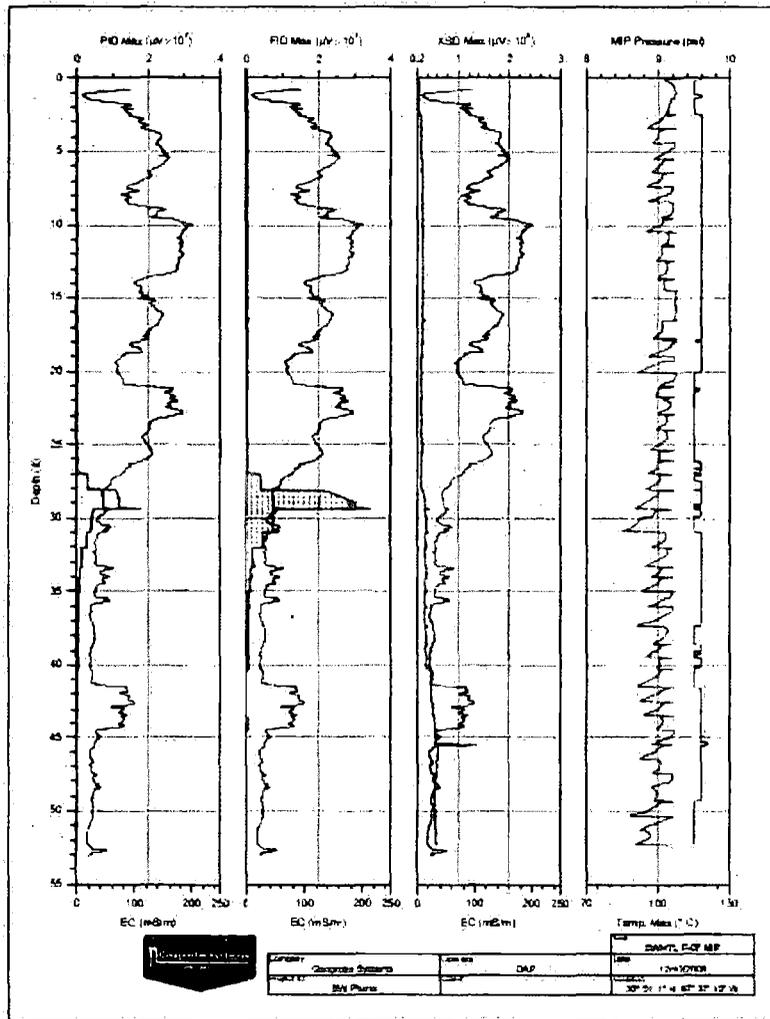
Response Tests using TCE

Pre or Post Log Response Test	Log ID:	PID Response 2.5 ppm	XSD Response 2.5 ppm	Log Footage	Membrane Footage
Pre-Log	MIP01	45,100	65,100	27	0
Pre-Log	MIP02	38,600	70,400	27	27
Pre-Log	MIP03	21,250	38,200	27	54
Pre-Log	MIP04	20,000	38,100	36	81
Pre-Log	MIP05	34,900	54,200	41	117
Pre-Log	MIP06	25,800	40,400	37	158
Pre-Log	MIP07	33,750	45,100	37	195
Pre-Log	MIP08	34,800	48,200	37	232
Pre-Log	MIP09	31,000	49,600	36	270
Post-Log	MIP09	29,400	42,700		306



APPENDIX IV

Sample Logs and Interpretation



Here is a MIP log showing the detectors (PID, FID and XSD) over the electrical conductivity graph as well as a graph of probe temperature and gas pressure.

The above log shows contamination from 27 ft to 33 ft bgs. The main detector response is on the PID and FID with minimal response on the XSD (Halogen Specific Detector). This indicates that the main contaminant would not contain halogenated (Cl-, Br-, FI-) atoms, but would be likely be hydrocarbon based. The contaminants are present in the lower electrical conductivity formations which typically are coarser grained, higher permeable formations. The increased temperature deflection of the MIP block heater around 25 ft provides an indication of where the water table may be in this log.

Detector Interpretation

Standard MIP systems are able to identify compound families and determine general compound classes. However the identification of individual compounds is not possible. Standard MIP systems have a continuous carrier gas flow that is brought to the detectors from the down-hole probe. To be able to effectively speciate (determine specific contaminant chemicals) the operator would need a highly modified system in place. The carrier gas stream would need to be trapped and run through either a mass spectrometry or secondary GC onsite.

Typical standard MIP configurations use 3 gas phase detectors: a photo-ionization detector (PID), flame-ionization (FID) and a halogen specific detector (XSD). The PID responds to compounds which have an ionization potential \leq electron voltage of the PID bulb. These compounds include both chlorinated and non-chlorinated hydrocarbons. A typical PID bulb has a 10.6eV lamp. The FID will respond when organic compounds (anything containing carbon) are present in the carrier gas stream in high enough concentration burn up in the flame which increases the flames ionization voltage. The XSD responds only to halogenated compounds which are made up of chlorinated (typical halogen environmental contaminant), brominated and fluorinated compounds. Based upon which detector or detector series a contaminant responds on, we can determine if the contaminants are halogenated or petroleum based.

Petroleum hydrocarbons will respond on the PID and FID but not on the XSD. Fresh gasoline primarily contains aromatic hydrocarbons such as benzene, toluene, ethyl benzene and xylenes, which respond strongly on a photo-ionization detector (PID) and not so well on the FID. As gasoline breaks down or weathers the molecular structure changes from primarily aromatic to mainly straight chain hydrocarbons (single bonded hydrocarbons). Straight chain hydrocarbons typically do not show up on the PID do having a higher ionization potential but will respond on a flame ionization detector (FID). Weathered petroleum will still have a decent signal on the PID but may show a stronger FID signal.

Chlorinated compounds such as trichloroethylene and perchloroethylene are detected by the XSD and PID and respond in a similar profile. This is typical of the common double bonded chlorinated compounds seen in the subsurface which have an ionization potential that the PID can see. Chlorinated compounds without multiple bonds such as chloroform, methylene chloride and 1,1,1-trichloroethane have an ionization potential higher than the PID electron voltage which results in a solid response on the XSD but will not show up on the PID.

The only sure way of determining contaminant concentration from MIP responses is to take confirmation soil and/or groundwater samples for laboratory analysis. After obtaining the results the actual concentrations can be compared to the MIP detector responses and concentrations may be estimated across the site.

APPENDIX V

GC1000 Configuration and Operating Parameters

GC1000 Configuration

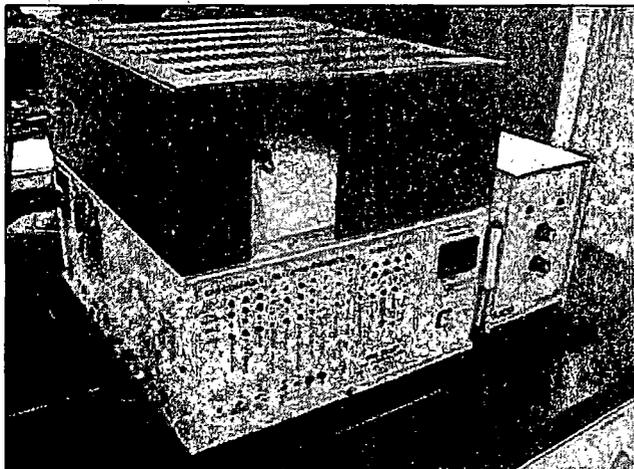


Figure 1: GC1000: SRI 310GC with XSD Controller.

SRI310 GC with PID, FID & OI Analytical
XSD (all standalone detectors)

Flows:

TL Carrier (N₂): 40ml/min
Detector split 60:40 – 24ml/min-XSD
16ml/min-FID

Nafion Dryer (installed in GC Oven)
80ml/min (2x carrier flow rate)

A built in air compressor is split underneath the GC between the XSD & FID. The XSD & FID air supply is controlled through the GC air pressure screw control on front of GC and with different air line sizes and lengths to provide 250ml/min to the FID and 30 ml/min to the XSD.

Detectors front of GC to back: XSD, FID & PID

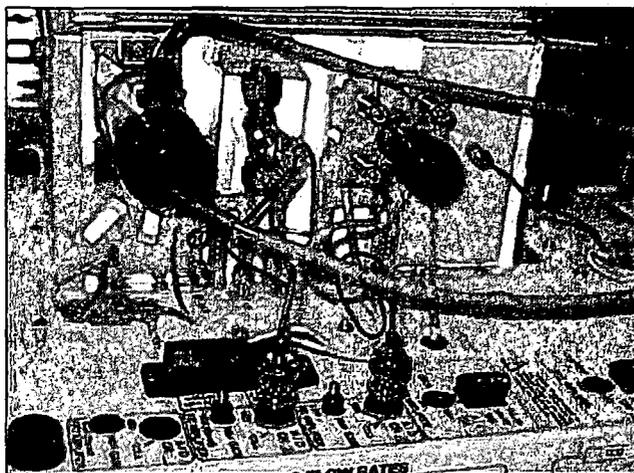


Figure 2: GC Detectors – left to right - XSD, FID, PID.

SRI 310 GC Detector 1 position – XSD
(not controlled by GC)

SRI 310 GC Detector 2 position – FID

SRI 310 GC Detector 3 position – PID

Nafion dryer installed inside GC oven

GC Oven set to 85°C – 130°C max temp.

Flow comes into the GC oven via a 1/16" bulkhead fitting located in the 4th detector position furthest back (upper right inside oven) behind the PID detector. The trunkline will connect to this bulkhead and a 1/16" stainless steel line transports flow into the Nafion dryer. Silco steel takes this to the PID lamp which is inserted up to the lamp and backed off a 1/16" and tightened. A 1/16" stainless steel line brings it back into the GC oven where it is split between the FID and XSD and sent to them via a silco-steel line to the XSD and a stainless steel line to the FID.

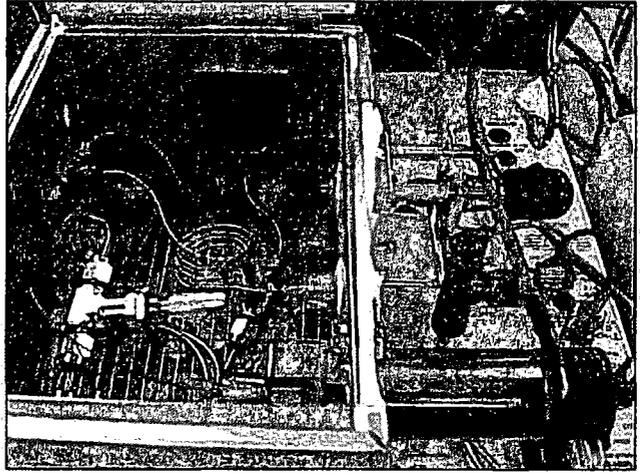


Figure 3: GC Oven Configuration.

Detector Operating Parameters:

PID:

- MIP Carrier Flow (N₂) – 100% - 40ml/min
- Carrier return back into oven split between XSD & FID
- Detector Temperature setting – 150°C
- PID current 70 (0.70ma)

FID:

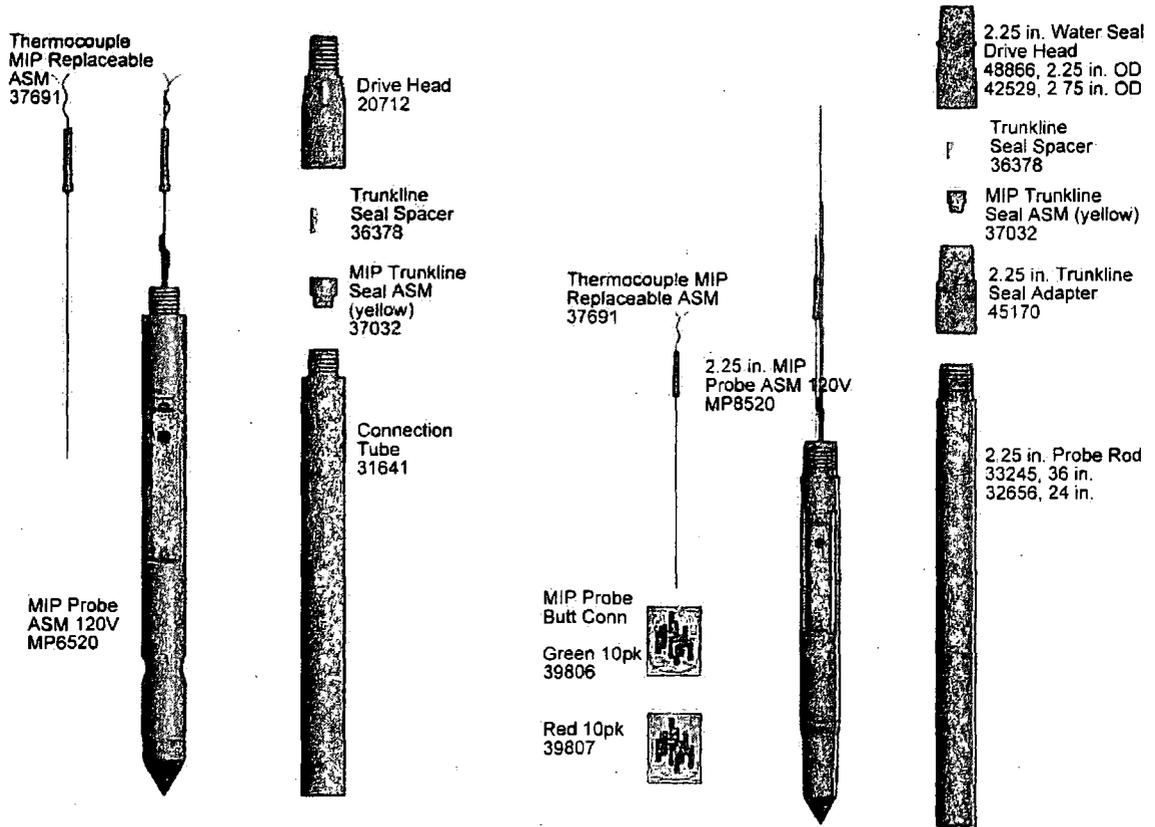
- Carrier N₂ MIP effluent – 40% - 16ml/min
- Hydrogen – 25ml/min
- AIR – 250ml/min
- Detector Temperature setting – 250°C
- FID igniter set at -600 (6.0V)

XSD:

- Carrier N₂ MIP effluent – 60% - 24ml/min
- Air – 30ml/min (split 50:50 wall & jet input of XSD)
- Detector Temperature setting – 1,100°C

APPENDIX VI

Tool Configurations



MIP - MP6520 Probe for 1.5 in. rods

MIP - MP8520 Probe for 2.25 in. rods

Equipment and tool specifications, including weights, dimensions, materials, and operating specifications included in this document are subject to change without notice. Where specifications are critical to your application, please consult Geoprobe Systems®.



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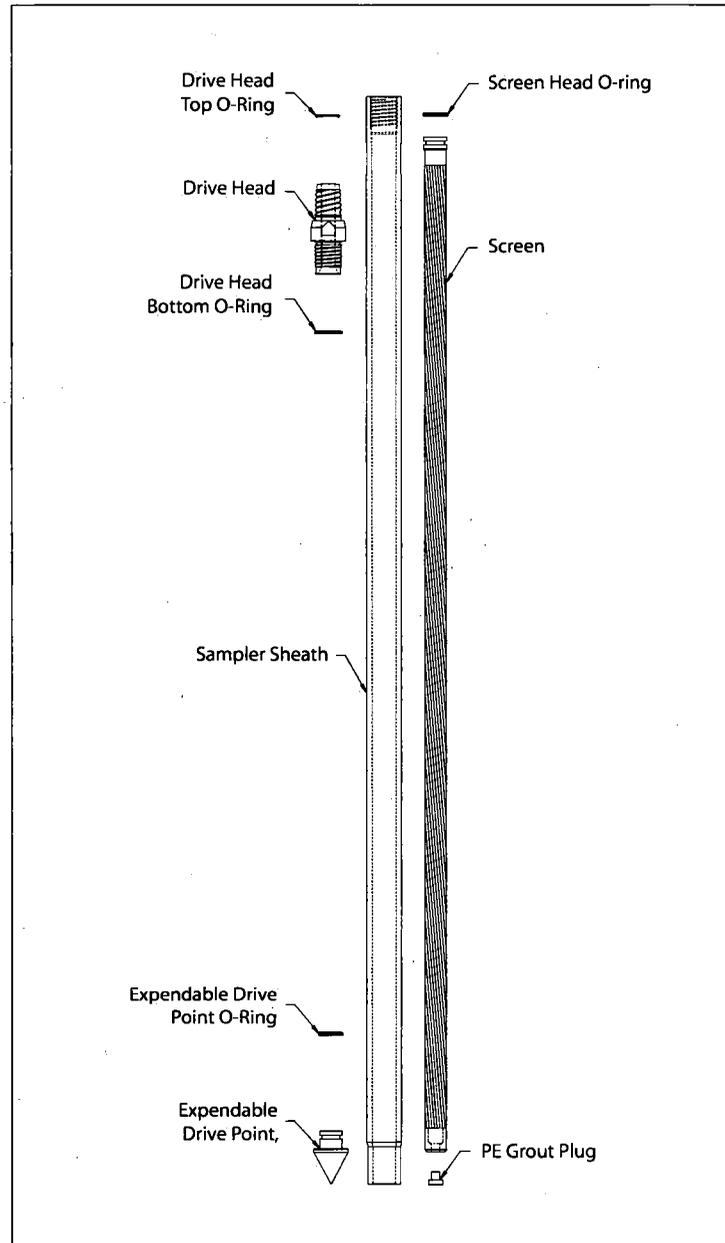
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GEOPROBE® SCREEN POINT 16 GROUNDWATER SAMPLER

STANDARD OPERATING PROCEDURE

Technical Bulletin No. MK3142

PREPARED: November, 2006



GEOPROBE® SCREEN POINT 16 GROUNDWATER SAMPLER PARTS



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**Screen Point 16 Groundwater Sampler is manufactured
under U.S. Patent 5,612,498**

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1.0 OBJECTIVE

The objective of this procedure is to drive a sealed stainless steel or PVC screen to depth, deploy the screen, obtain a representative water sample from the screen interval, and grout the probe hole during abandonment. The Screen Point 16 Groundwater Sampler enables the operator to conduct abandonment grouting that meets American Society for Testing and Materials (ASTM) Method D 5299 requirements for decommissioning wells and borings for environmental activities (ASTM 1993).

2.0 BACKGROUND

2.1 Definitions

Geoprobe®: A brand name of high quality, hydraulically powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and monitoring, soil conductivity and contaminant logging, grouting, and materials injection.

Screen Point 16 (SP16) Groundwater Sampler: A direct push device consisting of a PVC or stainless steel screen that is driven to depth within a sealed, steel sheath and then deployed for the collection of representative groundwater samples. The assembled SP16 Sampler is approximately 51.5 inches (1308 mm) long with an OD of 1.625 inches (41 mm). Upon deployment, up to 41 inches (1041 mm) of screen can be exposed to the formation. The Screen Point 16 Groundwater Sampler is designed for use with 1.5-inch probe rods and machines equipped with the more powerful GH60 Hydraulic Hammer. Operators with GH40 Series hammers may chose to use this sampler in soils where driving is difficult.

Rod Grip Pull System: An attachment mounted on the hydraulic hammer of a direct push machine which makes it possible to retract the tool string with extension rods or flexible tubing protruding from the top of the probe rods. The Rod Grip Pull System includes a pull block with rod grip jaws that are bolted directly to the machine. A removable handle assembly straddles the tool string while hooking onto the pull block to effectively grip the probe rods as the hammer is raised. A separate handle assembly is required for each probe rod diameter.

2.2 Discussion

In this procedure, the assembled Screen Point 16 Groundwater Sampler (Fig. 2.1A) is threaded onto the leading end of a Geoprobe® probe rod and advanced into the subsurface with a Geoprobe® direct push machine. Additional probe rods are added incrementally and advanced until the desired sampling interval is reached. While the sampler is advanced to depth, O-ring seals at each rod joint, the drive head, and the expendable drive point provide a watertight system. This system eliminates the threat of formation fluids entering the screen before deployment and assures sample integrity.

Once at the desired sampling interval, extension rods are sent downhole until the leading rod contacts the bottom of the sampler screen. The tool string is then retracted approximately 44 inches (1118 mm) while the screen is held in place with the extension rods (Fig. 2.1B). As the tool string is retracted, the expendable point is released from the sampler sheath. The tool string and sheath may be retracted the full length of the screen or as little as a few inches if a small sampling interval is desired.

There are three types of screens that can be used in the Screen Point 16 Groundwater Sampler. Two of these, a stainless steel screen with a standard slot size of 0.004 inches (0.10 mm) and a PVC screen with a standard slot size of 0.010 inches (0.25 mm), are recovered with the tool string after sampling. The third screen is also manufactured from PVC with a standard slot size of 0.010 inches (0.25 mm), but is designed to be left downhole when sampling is complete. This disposable screen has an exposed screen length of approximately 43 inches (1092 mm). The two screens that are recovered with the sampler both have an exposed screen length of approximately 41 inches (1041 mm).

(continued on following page)

An O-ring on the head of the stainless steel screens maintains a seal at the top of the screen. As a result, any liquid entering the sampler during screen deployment must first pass through the screen. PVC screens do not require an O-ring because the tolerance between the screen head and sampler sheath is near that of the screen slot size.

The screens are constructed such that flexible tubing, a mini-bailer, or a small-diameter bladder pump can be inserted into the screen cavity. This makes direct sampling possible from anywhere within the saturated zone. A removable plug in the lower end of the screens allows the user to grout as the sampler is extracted for further use.

Groundwater samples can be obtained in a number of ways. A common method utilizes polyethylene (TB25L) or Teflon® (TB25T) tubing and a Check Valve Assembly (GW4210). The check valve (with check ball) is attached to one end of the tubing and inserted down the casing until it is immersed in groundwater. Water is pumped through the tubing and to the ground surface by oscillating the tubing up and down.

An alternative means of collecting groundwater samples is to attach a peristaltic or vacuum pump to the tubing. This method is limited in that water can be pumped to the surface from a maximum depth of approximately 26 feet (8 m). Another technique for groundwater sampling is to use a stainless steel Mini-Bailer Assembly (GW41). The mini-bailer is lowered down the inside of the casing below the water level where it fills with water and is then retrieved from the casing.

The latest option for collecting groundwater from the SP16 sampler is to utilize a Geoprobe® MB470 Series Mechanical Bladder Pump (MBP)*. The MBP may be used to meet requirements of the low-flow sampling protocol (Puls and Barcelona 1996, ASTM 2003). Through participation in a U.S. EPA Environmental Technology Verification study, it was confirmed that the MB470 can provide representative samples (EPA 2003).

**The Mechanical Bladder Pump is manufactured under U.S. Patent No. 6,877,965 issued April 12, 2005.*

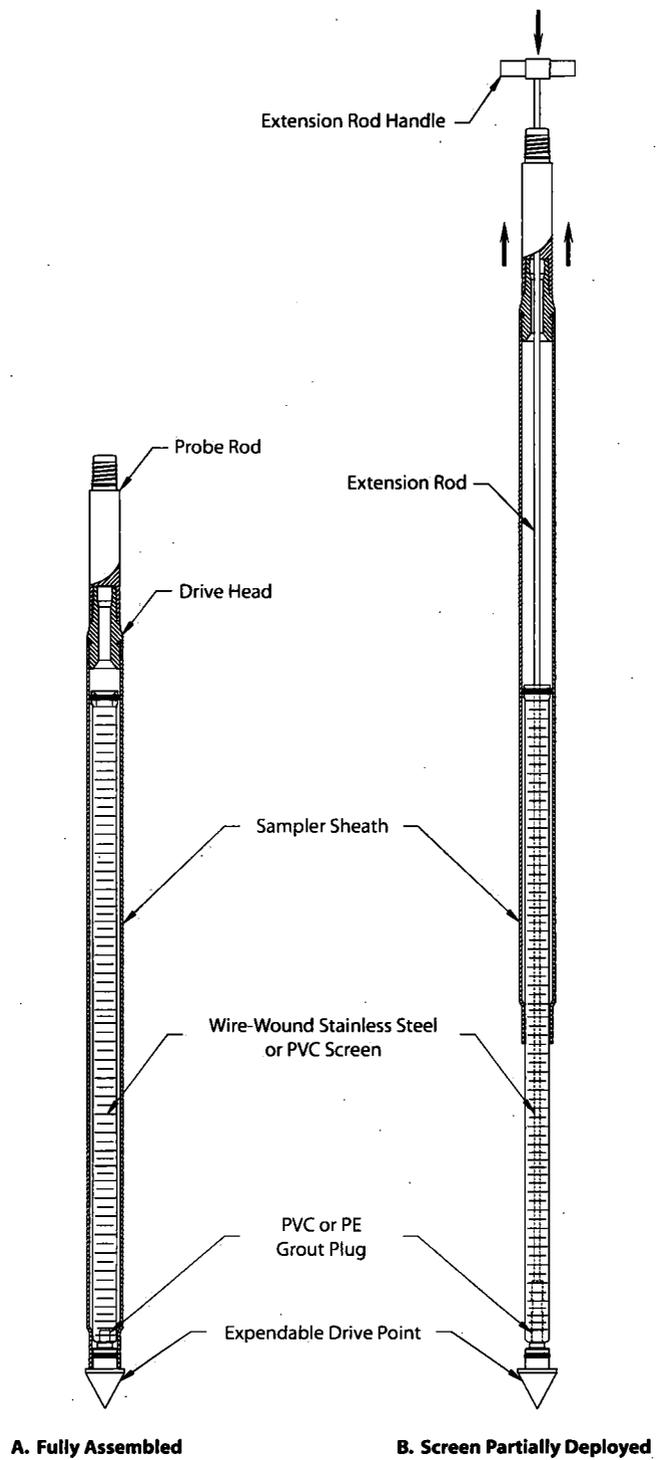


FIGURE 2.1
Screen Point 16 Groundwater Sampler

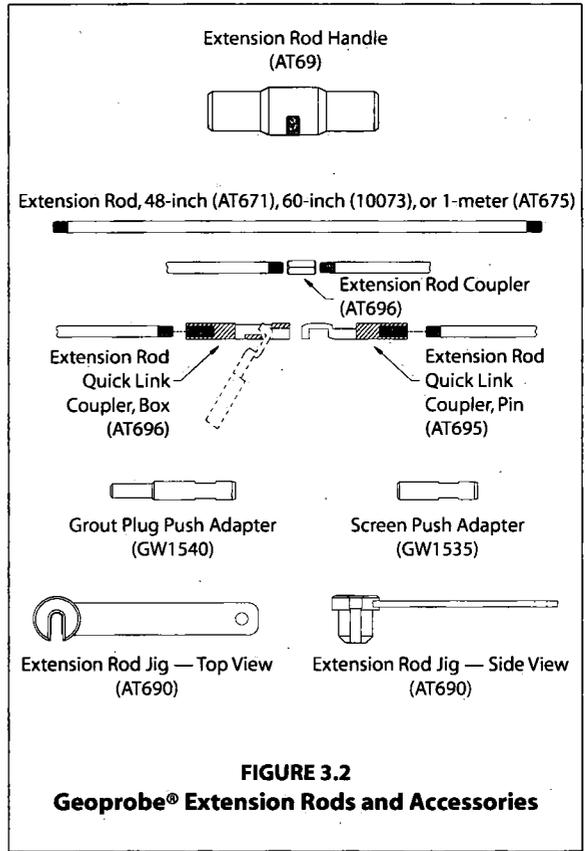
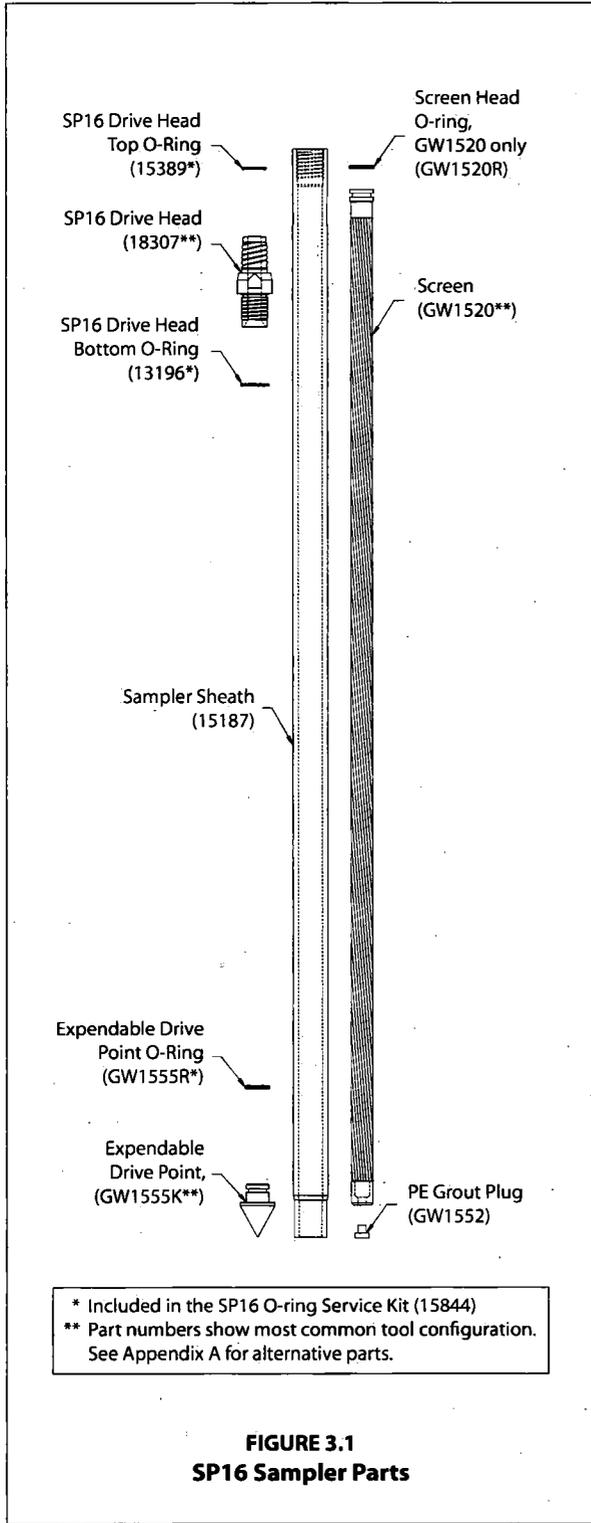
3.0 TOOLS AND EQUIPMENT

The following tools and equipment can be used to successfully recover representative groundwater samples with the Geoprobe® Screen Point 16 Groundwater Sampler. Refer to Figures 3.1 and 3.2 for identification of the specified parts. Tools are listed below for the most common SP16 / 1.5-inch probe rod configurations. Additional parts for optional rod sizes and accessories are listed in Appendix A.

SP16 Sampler Parts	Part Number
SP16 Sampler Sheath.....	15187
SP16 Drive Head, 0.5-inch bore, 1.5-inch rods*	18307
SP16 O-ring Service Kit, 1.5-inch rods (<i>includes 4 each of the O-ring packets below</i>)	15844
<i>O-rings for Top of SP16 Drive Head, 1.5-inch rods only (Pkt. of 25)</i>	15389
<i>O-rings for Bottom of SP16 Drive Head (Pkt. of 25)</i>	13196
<i>O-rings for GW1520 Screen Head (Pkt. of 25)</i>	GW1520R
<i>O-rings for SP16 Expendable Drive Point (Pkt. of 25)</i>	GW1555R
Screen, Wire-Wound Stainless Steel, 4-Slot*	GW1520
Grout Plugs, PE (Pkg. of 25)	GW1552K
Expendable Drive Points, steel, 1.625-inch OD (Pkg. of 25)*	GW1555K
Screen Point 16 Groundwater Sampler Kit, 1.5-inch Probe Rods (<i>includes 1 each of:</i> 15187, 18307, 15844, GW1520, GW1535, GW1540, GW1555K, and GW1552K).....	15770
Probe Rods and Probe Rod Accessories	Part Number
Drive Cap, 1.5-inch probe rods, threadless, (for GH60 Hammer).....	12787
Pull Cap, 1.5-inch probe rods	15090
Probe Rod, 1.5-inch x 60-inch*	11121
Extension Rods and Extension Rod Accessories	Part Number
Screen Push Adapter.....	GW1535
Grout Plug Push Adapter.....	GW1540
Extension Rod, 60-inch*	10073
Extension Rod Coupler.....	AT68
Extension Rod Handle	AT69
Extension Rod Jig.....	AT690
Extension Rod Quick Link Coupler, pin.....	AT695
Extension Rod Quick Link Coupler, box.....	AT696
Grout Accessories	Part Number
Grout Nozzle, for 0.375-inch OD tubing.....	GW1545
High-Pressure Nylon Tubing, 0.375-inch OD / 0.25-inch ID, 100-ft. (30 m).....	11633
Grout Machine, self-contained*	GS1000
Grout System Accessories Package, 1.5-inch rods	GS1015
Groundwater Purging and Sampling Accessories	Part Number
Polyethylene Tubing, 0.375-inch OD, 500 ft.*	TB25L
Check Valve Assembly, 0.375-inch OD Tubing*	GW4210
Water Level Meter, 0.438-inch OD Probe, 100 ft. cable*	GW2000
Mechanical Bladder Pump**	MB470
Mini Bailer Assembly, stainless steel	GW41
Additional Tools	Part Number
Adjustable Wrench, 6.0-inch	FA200
Adjustable Wrench, 10.0-inch	FA201
Pipe Wrenches	NA

* See Appendix A for additional tooling options.

** Refer to the Standard Operating Procedure (SOP) for the Mechanical Bladder Pump (Technical Bulletin No. MK3013) for additional tooling needs.



4.0 OPERATION

4.1 Basic Operation

The SP16 sampler utilize a stainless steel or PVC screen which is encased in an alloy steel sampler sheath. An expendable drive point is placed in the lower end of the sheath while a drive head is attached to the top. O-rings on the drive head and expendable point provide a watertight sheath which keeps contaminants out of the system as the sampler is driven to depth.

Once the sampling interval is reached, extension rods equipped with a screen push adapter are inserted down the ID of the probe rods. The tool string is then retracted up to 44 inches (1118 mm) while the screen is held in place with the extension rods. The system is now ready for groundwater sampling. When sampling is complete, a removable plug in the bottom of the screen allows for grouting below the sampler as the tool string is retrieved.

4.2 Sampler Options

The Screen Point 15 and Screen Point 16 Groundwater Samplers are nearly identical. Subtle differences in the design of the SP16 sampler make it more durable than the earlier SP15 system. Operators of GH60-equipped machines should always utilize SP16 tooling. Operators of machines equipped with GH40 Series hammers may also choose SP16 tooling when sampling in difficult probing conditions.

A 1.75-inch OD Expendable Drive Point (17066K) and Disposable PVC Screen (16089) provide two useful options for the SP16 sampler. The 1.75-inch drive point may be used when soil conditions make it difficult to remove the sampler after driving to depth. The disposable PVC screen may be left downhole after sampling (when regulations permit) to eliminate the time required for screen decontamination.

4.3 Decontamination

In order to collect representative groundwater samples, all sampler parts must be thoroughly cleaned before and after each use. Scrub all metal parts using a stiff brush and a nonphosphate soap solution. Steam cleaning may be substituted for hand-washing if available. Rinse with distilled water and allow to air-dry before assembly.

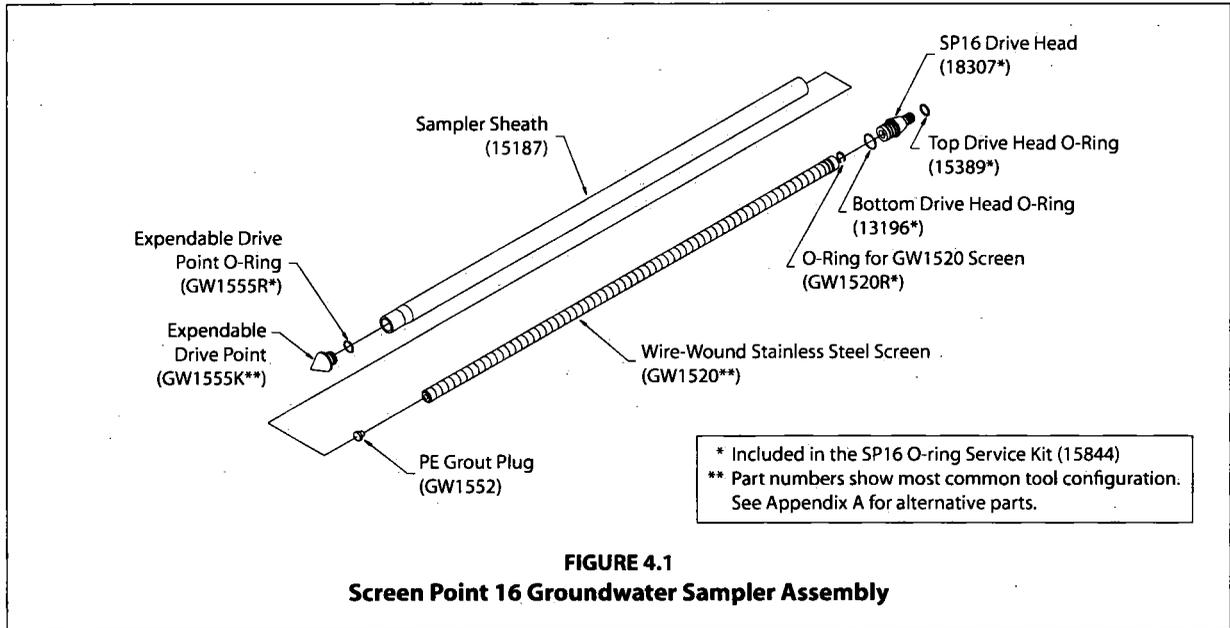
4.4 SP16 Sampler Assembly (Figure 4.1)

Part numbers are listed for a standard SP16 sampler using 1.5-inch probe rods. Refer to Page 6 for screen and drive head alternatives.

1. Place an O-ring on a steel expendable drive point (GW1555K). Firmly seat the expendable point in the necked end of a sampler sheath (15187).
2. Install a PE Grout Plug (GW1552) in the bottom end of a Wire-wound Stainless Steel Screen (GW1520). Place a GW1520R O-ring in the groove on the top end of the screen.
3. Slide the screen inside of the sampler sheath with the grout plug toward the bottom of the sampler. Ensure that the expendable point was not displaced by the screen.
4. Install a bottom O-ring (13196) on a Drive Head (18307 or 15188). Thread the drive head into the sampler sheath using an adjustable wrench if necessary to ensure complete engagement of the threads. Attach a Drive Cap (12787 or 15590) to the top of the drive head.

NOTE: The 18307 drive head should be used whenever possible as the smaller 0.5-inch ID provides a greater material cross-section for increased durability.

Sampler assembly is complete.



4.5 Advancing the SP16 Sampler

To provide adequate room for screen deployment with the Rod Grip Pull System, the probe derrick should be extended a little over halfway out of the carrier vehicle when positioning for operation.

1. Begin by placing the assembled sampler (Fig. 2.1.A) in the driving position beneath the hydraulic hammer of the direct push machine as shown in Figure 4.2.
2. Advance the sampler with the throttle control at slow speed for the first few feet to ensure that the sampler is aligned properly. Switch to fast speed for the remainder of the probe stroke.

3. Completely raise the hammer assembly. Remove the drive cap and place an O-ring in the top groove of the drive head. Distilled water may be used to lubricate the O-ring if needed.

Add a probe rod (length to be determined by operator) and reattach the drive cap to the rod string. Drive the sampler the entire length of the new rod with the throttle control at fast speed.

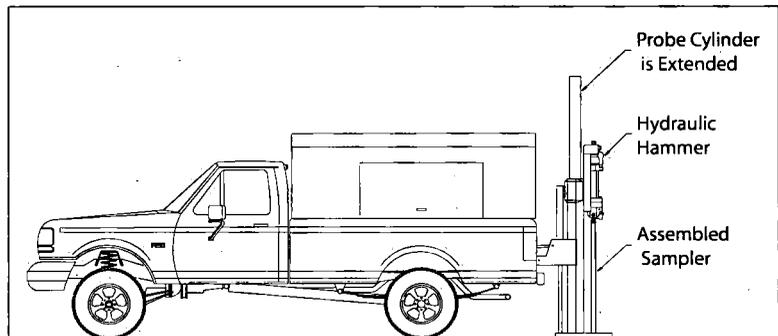


FIGURE 4.2
Screen Point 16 Groundwater Sampler in Driving Position

4. Repeat Step 3 until the desired sampling interval is reached. Approximately 12 inches (305 mm) of the last probe rod must extend above the ground surface to allow attachment of the puller assembly. A 12-inch (305 mm) rod may be added if the tool string is over-driven.
5. Remove the drive cap and retract the probe derrick away from the tool string.

4.6 Screen Deployment

1. Thread a screen push adapter (GW1535) on an extension rod of suitable length (AT671, 10073, or AT675). Attach a threaded coupler (AT68) to the other end of the extension rod. Lower the extension rod inside of the probe rod taking care not to drop it down the tool string. An extension rod jig (AT690) may be used to hold the rods.
2. Add extension rods until the adapter contacts the bottom of the screen. To speed up this step, it is recommended that Extension Rod Quick Links (AT695 and AT696) are used at every other rod joint.
3. Ensure that at least 48 inches (1219 mm) of extension rod protrudes from the probe rod. Thread an extension rod handle (AT69) on the top extension rod.
4. Maneuver the probe assembly into position for pulling.
5. Raise (pull) the tool string while physically holding the screen in place with the extension rods (Fig. 4.3.B). A slight knock with the extension rod string will help to dislodge the expendable point and start the screen moving inside the sheath.

Raise the hammer and tool string about 44 inches (1118 cm) if using a GW1520 or GW1530 screen. At this point the screen head will contact the necked portion of the sampler sheath (Fig. 4.3.C.) and the extension rods will rise with the probe rods. Use care when deploying a PVC screen so as not to break the screen when it contacts the bottom of the sampler sheath.

The Disposable Screen (16089) will extend completely out of the sheath if the tool string is raised more than 45 inches (1143 mm). Measure and mark this distance on the top extension rod to avoid losing the screen during deployment.

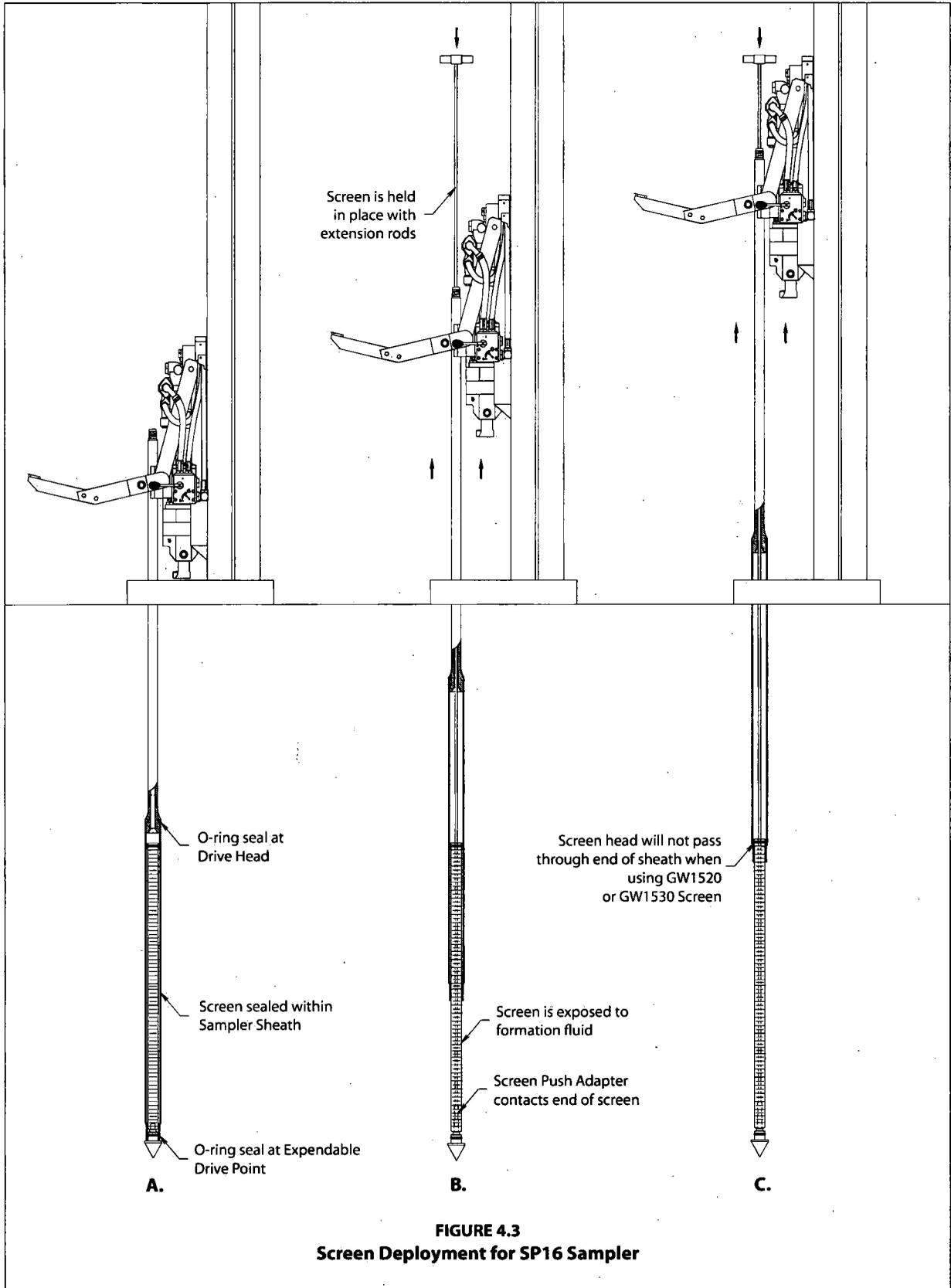
6. Remove the rod grip handle, lower the hammer assembly, and retract the probe derrick. Remove the top extension rod (with handle) and top probe rod. Finally, extract all extension rods.
7. Groundwater samples can now be collected with a mini-bailer, peristaltic or vacuum pump, tubing bottom check valve assembly, bladder pump, or other acceptable small diameter sampling device.

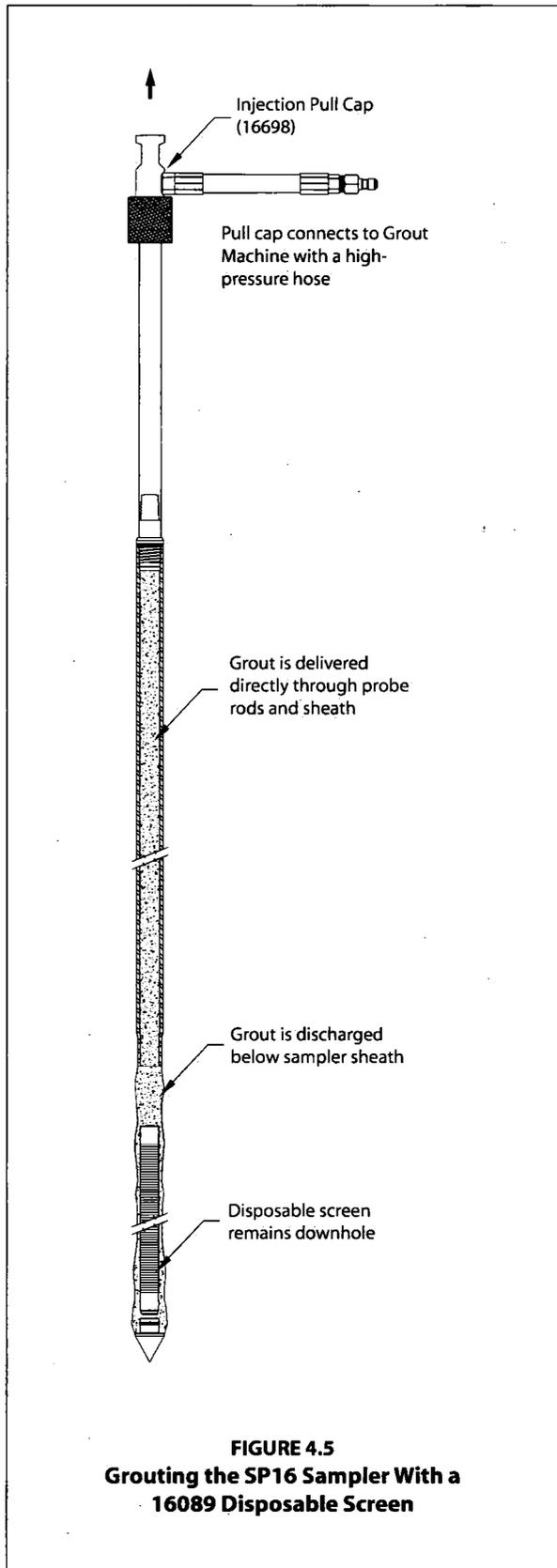
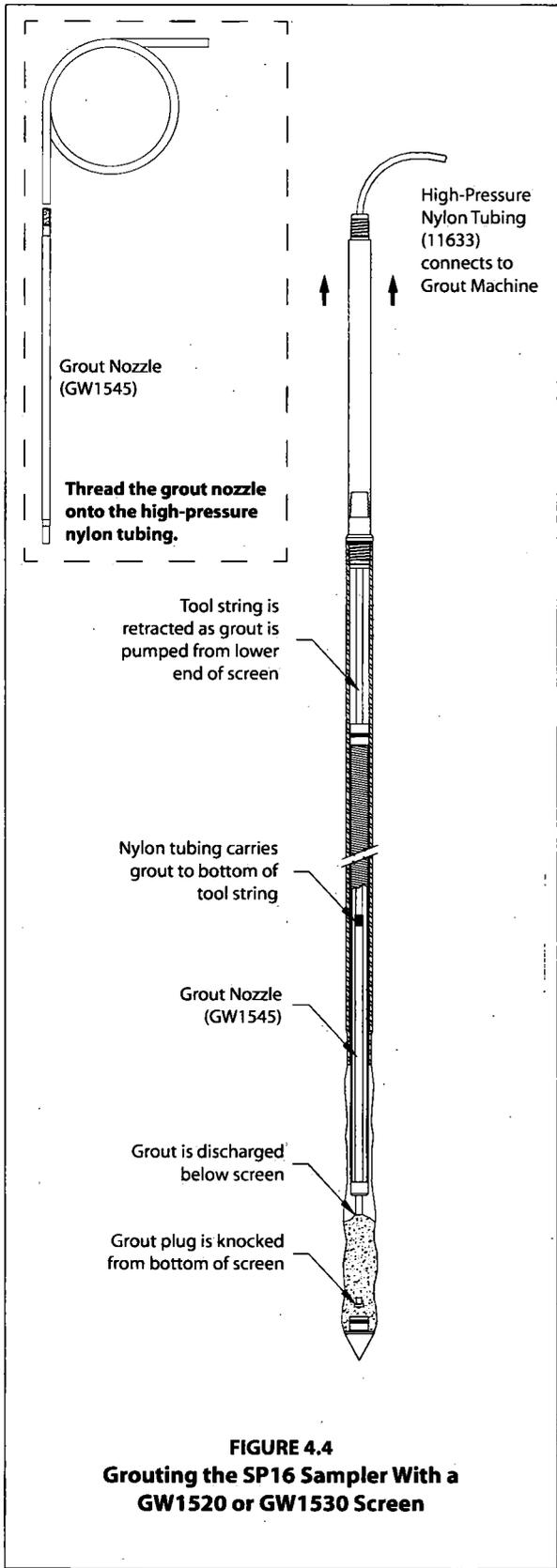
When inserting tubing or a bladder pump down the rod string, ensure that it enters the screen interval. The leading end of the tubing or bladder pump will sometimes catch at the screen head giving the illusion that the bottom of the screen has been reached. An up-and-down motion combined with rotation helps move the tubing or bladder pump past the lip and into the screen.

4.7 Abandonment Grouting for GW1520 and GW1530 Screens

The SP16 Sampler can meet ASTM D 5299 requirements for abandoning environmental wells or borings when grouting is conducted properly. A removable grout plug makes it possible to deploy tubing through the bottom of GW1520 and GW1530 screens. A GS500 or GS1000 Grout Machine is then used to pump grout into the open probe hole as the sampler is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations.

1. Maneuver the probe assembly into position for pulling. Attach the rod grip puller to the top probe rod. Raise the tool string approximately 4 to 6 inches (102 to 152 cm) to allow removal of the grout plug.
2. Thread the Grout Plug Push Adapter (GW1540) onto an extension rod. Insert the adapter and extension rod inside the probe rod string. Add extension rods until the adapter contacts the grout plug at the bottom of the screen. Attach the handle to the top extension rod. When the extension rods are slightly raised and lowered, a relatively soft rebound should be felt as the adapter contacts the grout plug. This is especially true when using a PVC screen.





3. Place a mark on the extension rod even with the top of the probe rod. Apply downward pressure on the extension rods and push the grout plug out of the screen. The mark placed on the extension rod should now be below the top of the probe rod. Remove all extension rods.

Note: When working with a stainless steel screen, it may be necessary to raise and quickly lower the extension rods to jar the grout plug free. When the plug is successfully removed, a metal-on-metal sensation may be noted as the extension rods are gently "bounced" within the probe rods.

4. A Grout Nozzle (GW1545) is now connected to High-Pressure Nylon Tubing (11633) and inserted down through the probe rods to the bottom of the screen (Fig. 4.4). It may be necessary to pump a small amount of clean water through the tubing during deployment to jet out sediments that settled in the bottom of the screen. Resistance will sometimes be felt as the grout nozzle passes through the drive head. Rotate the tubing while moving it up-and-down to ensure that the nozzle has reached the bottom of the screen and is not hung up on the drive head.

Note: All probe rods remain strung on the tubing as the tool string is pulled. Provide extra tubing length to allow sufficient room to lay the rods on the ground as they are removed. An additional 20 feet is generally enough.

5. Operate the grout pump while pulling the first rod with the rod grip pull system. Coordinate pumping and pulling rates so that grout fills the void left by the sampler. After pulling the first rod, release the rod grip handle, fully lower the hammer, and regrip the tool string. Unthread the top probe and slide it over the tubing placing it on the ground near the end of the tubing.
6. Repeat Step 5 until the sampler is retrieved. Do not bend or kink the tubing when pulling and laying out the probe rods. Sharp bends create weak spots in the tubing which may burst when pumping grout. Remember to operate the grout pump only when pulling the rod string. The probe hole is thus filled with grout from the bottom up as the rods are extracted.
7. Promptly clean all probe rods and sampler parts before the grout sets up and clogs the equipment.

4.8 Abandonment Grouting for the 16089 Disposable Screen

ASTM D 5299 requirements can also be met for the SP16 samplers when using the 16089 disposable screen. Because the screen remains downhole after sampling, the operator may choose either to deliver grout to the bottom of the tool string with nylon tubing or pump grout directly through the probe rods using an Injection Pull Cap (16698). A GS500 or GS1000 Grout Machine is needed to pump grout into the open probe hole as the sampler is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations.

1. Maneuver the probe assembly into position for pulling with the rod grip puller.
2. Thread the screen push adapter onto an extension rod. Insert the adapter and extension rod inside the probe rod string. Add extension rods until the adapter contacts the bottom of the screen. Attach the handle to the top extension rod.
3. The disposable screen must be extended at least 46 inches (1168 mm) to clear the bottom of the sampler sheath. Considering the length of screen deployed in Section 4.7, determine the remaining distance required to fully extend the screen from the sheath. Mark this distance on the top extension rod.
4. Pull the tool string up to the mark on the top extension rod while holding the disposable screen in place.

The screen is now fully deployed and the sampler is ready for abandonment grouting. Apply grout to the bottom of the tool string during retrieval using either flexible tubing (as described in Section 4.7) or an injection pull cap (Fig. 4.5). This section continues with a description of grouting with a pull cap.

5. Remove the rod grip handle and maneuver the probe assembly directly over the tool string. Thread an Injection Pull Cap (16698) onto the top probe rod and close the hammer pull latch over the top of the pull cap.
6. Connect the pull cap to a Geoprobe® grout machine using a high-pressure grout hose.
7. Operate the pump to fill the entire tool string with grout. When a sufficient volume has been pumped to fill the tool string, begin pulling the rods and sampler while continuing to operate the grout pump. Considering the known pump volume and sampler cross-section, time tooling withdrawal to slightly "overpump" grout into the subsurface. This will ensure that all voids are filled during sampler retrieval.

The grouting process can lubricate the probe hole sufficiently to cause the tool string to slide back downhole when disconnected from the pull cap. Prevent this by withdrawing the tool string with the rod grip puller while maintaining a connection to the grout machine with the pull cap.

4.9 Retrieving the Screen Point 16 Sampler

If grouting is not required, the Screen Point 16 Sampler can be retrieved by pulling the probe rods as with most other Geoprobe® applications. The Rod Grip Pull System should be used for this process as it allows the operator to remove rods without completely releasing the tool string. This avoids having the probe rods fall back downhole when released during the pulling procedure. A standard Pull Cap (15164) may still be used if preferred. Refer to the Owner's Manual for your Geoprobe® direct push machine for specific instructions on pulling the tool string.

5.0 REFERENCES

- American Society of Testing and Materials (ASTM), 2003. D6771-02 Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations. ASTM, West Conshohocken, PA. (www.astm.org)
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Appendix A ALTERNATIVE PARTS

The following parts are available to meet unique soil conditions. See section 3.0 for a complete listing of the common tool configurations for the Geoprobe® Screen Point 16 Groundwater Sampler.

SP16 Sampler Parts and Accessories.....	Part Number
SP16 Drive Head, 0.625-inch bore, 1.5-inch rods.....	15188
Expendable Drive Points, aluminum, 1.625-inch OD (Pkg. of 25).....	GW1555ALK
Expendable Drive Points, steel, 1.75-inch OD (Pkg. of 25).....	17066K
Screen, PVC, 10-Slot.....	GW1530
Screen, Disposable, PVC, 10-Slot.....	16089
Groundwater Purging and Sampling Accessories	Part Number
Polyethylene Tubing, 0.25-inch OD, 500 ft.....	TB17L
Polyethylene Tubing, 0.5-inch OD, 500 ft.....	TB37L
Polyethylene Tubing, 0.625-inch OD, 50 ft.....	TB50L
Check Valve Assembly, 0.25-inch OD Tubing.....	GW4240
Check Valve Assembly, 0.5-inch OD Tubing.....	GW4220
Check Valve Assembly, 0.625-inch OD Tubing.....	GW4230
Water Level Meter, 0.375-inch OD Probe, 100-ft. cable.....	GW2001
Water Level Meter, 0.438-inch OD Probe, 200-ft. cable.....	GW2002
Water Level Meter, 0.375-inch OD Probe, 200-ft. cable.....	GW2003
Water Level Meter, 0.438-inch OD Probe, 30-m cable.....	GW2005
Water Level Meter, 0.438-inch OD Probe, 60-m cable.....	GW2007
Water Level Meter, 0.375-inch OD Probe, 60-m cable.....	GE2008
Grouting Accessories.....	Part Number
Grout Machine, auxiliary-powered.....	GS500
Probe Rods, Extension Rods, and Accessories	Part Number
Probe Rod, 1.5-inch x 1-meter.....	17899
Probe Rod, 1.5-inch x 48-inch.....	13359
Drive Cap, 1.5-inch rods (for GH40 Series Hammer).....	15590
Rod Grip Pull Handle, 1.5-inch Probe Rods (for GH40 Series Hammer).....	GH1555
Extension Rod, 48-inch.....	AT671
Extension Rod, 1-meter.....	AT675

Equipment and tool specifications, including weights, dimensions, materials, and operating specifications included in this brochure are subject to change without notice. Where specifications are critical to your application, please consult Geoprobe Systems®.



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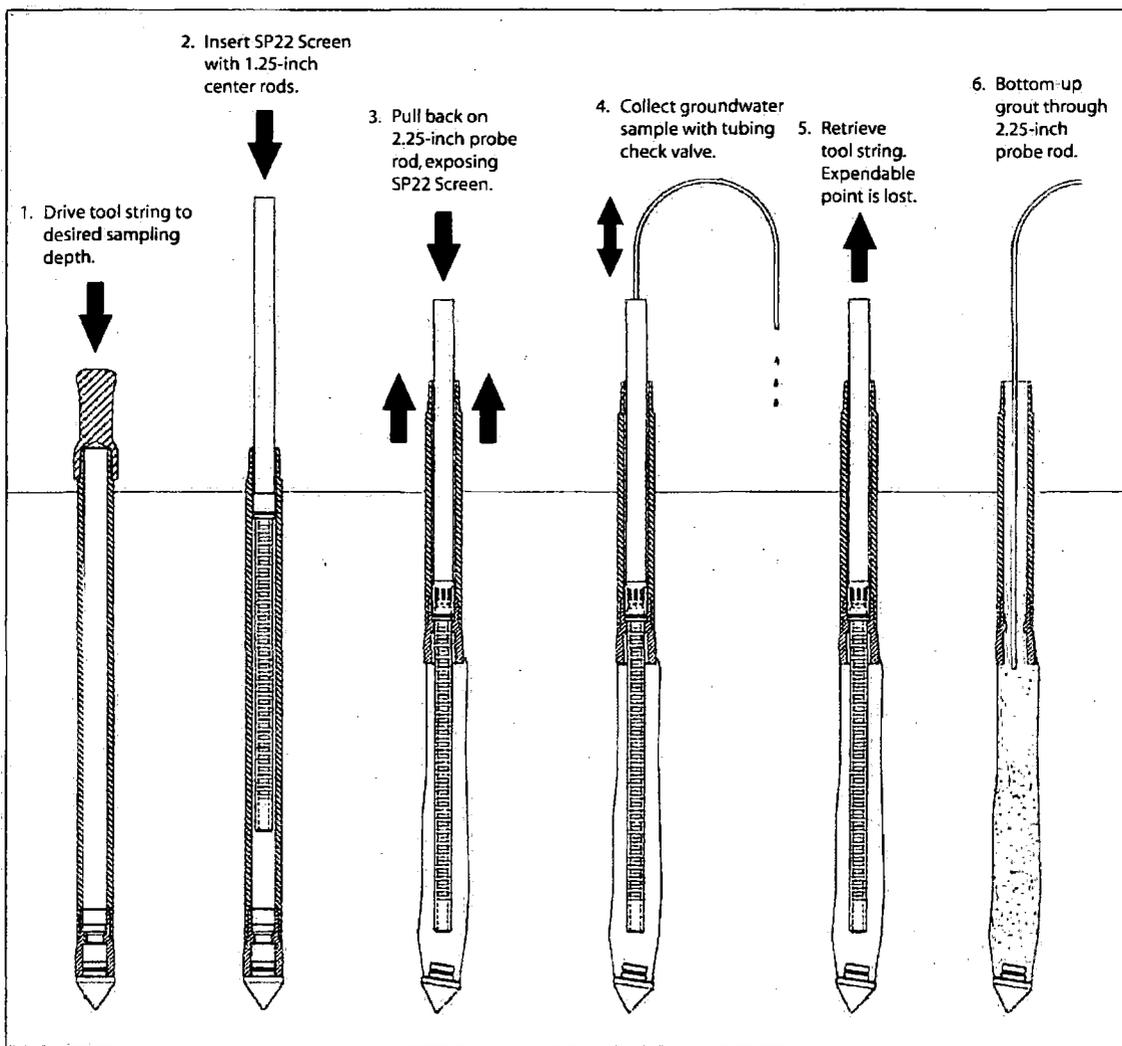
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GEOPROBE® SCREEN POINT 22 GROUNDWATER SAMPLER

STANDARD OPERATING PROCEDURE

Technical Bulletin No. MK3173

PREPARED: April 2010



OPERATION OF THE GEOPROBE® SCREEN POINT 22 GROUNDWATER SAMPLER



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**Screen Point 22 Groundwater Sampler is manufactured
under U.S. Patent 5,612,498**

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1.0 OBJECTIVE

The objective of this procedure is to deploy a stainless steel or PVC screen at depth, obtain a representative water sample from the screen interval, and grout the probe hole during abandonment. The Screen Point 22 Groundwater Sampler enables the operator to conduct abandonment grouting that meets American Society for Testing and Materials (ASTM) Method D 5299 requirements for decommissioning wells and borings for environmental activities (ASTM 1993).

2.0 BACKGROUND

2.1 Definitions

Geoprobe®: A brand name of high quality, hydraulically powered machines that utilize static force and percussion or rotation to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform activities such as soil core and soil gas sampling, groundwater sampling and monitoring, soil conductivity and contaminant logging, grouting, and materials injection.

Screen Point 22 (SP22) Groundwater Sampler: A direct push device consisting of a PVC or stainless steel screen that is lowered (post-run) to depth within a sealed string of steel probe rods and then deployed for the collection of representative groundwater samples. Upon deployment, up to 48 inches (1219 mm) of screen can be exposed to the formation. There is also an optional 12-inch screen that can be used. The Screen Point 22 Groundwater Sampler is designed for use with 2.25-inch probe rods and machines equipped with the more powerful GH60 and GH80 series hydraulic hammers. Operators with GH40 series hammers may choose to use this sampler in soils where driving is easier.

Rod Grip Pull System: An attachment mounted on the hydraulic hammer of a direct push machine which makes it possible to retract the tool string with probe rods or flexible tubing protruding from the top of the probe rods. The Rod Grip Pull System includes a pull block with rod grip jaws that are bolted directly to the machine. A removable handle assembly straddles the tool string while hooking onto the pull block to effectively grip the probe rods as the hammer is raised. A separate handle assembly is required for each probe rod diameter.

2.2 Discussion (Fig. 2.1)

In this procedure, 2.25-inch probe rods are advanced into the subsurface with a Geoprobe® subsurface machine (Fig. 2.1, Step 1). While the tool string is advanced to depth, O-ring seals at each rod joint, the expendable point holder, and the expendable drive point provide a watertight system. This eliminates the threat of formation fluids entering the screen before deployment and assures sample integrity.

Once the leading end of the 2.25-inch probe rods reaches the desired sampling interval, an SP22 screen is lowered to the bottom of the rods using a string of either 1.25-inch outside diameter (OD) light-weight center rods, 1.25-inch probe rods, or 0.75-inch schedule 40 flush-thread PVC riser (Fig. 2.1, Step 2). The 2.25-inch rods are then retracted while the SP22 screen is held in place with the 1.25-inch rods or PVC riser (Fig. 2.1, Step 3). As the 2.25-inch tool string is retracted, the expendable point is released from the expendable point holder. The tool string and expendable point holder may be retracted the full length of the screen or as little as a few inches if a small sampling interval is desired.

The SP22 Sampler can also be used with the Geoprobe® DT22 system. (Fig. 2.2)

(continued on following page)

Expendable Drive Points

The SP22 system utilizes an SP22 Expendable Point Holder (33764) and standard 2.45-inch (62-mm) OD steel Expendable Drive Points for 2.25-inch probe rods (AT2015K). Extended Shank Expendable Drive Points (19442) are available for soft soil conditions where standard points may be advanced out of the point holder during percussion. A third option is to use a part number 43128 SP22 Expendable Point Holder along with 1.625-inch (41-mm) steel Expendable Drive Points (GW1555K). These smaller drive points are more economical to purchase and ship, but must not be used with GH80 Series Hydraulic Hammers as they may not stay seated during percussion.

Screens

Two types of screens have been developed for use in the Screen Point 22 Groundwater Sampler - a stainless steel screen with a standard slot size of 0.004 inches (0.10 mm) and a PVC screen with a standard slot size of 0.010 inches (0.25 mm). These screens are available in nominal 48- and 12-inch lengths. Effective screen lengths for the 48- and 12-inch PVC screens are 48 inches (1219 mm) and 12 inches (305 mm), while 48- and 12-inch stainless steel screens have effective screen lengths of 43 inches (1092 mm) and 14 inches (356 mm) respectively. Both types of screens are recovered with the tool string after sampling.

The SP22 PVC Screen Head Adapter (37871) provides yet another screen option for the SP22 sampler. Using this adapter, a section of slotted 0.75-inch Schedule 40 PVC pipe may be lowered through the 2.25-inch probe rods using a string of flush-threaded 0.75-inch Schedule 40 PVC Riser. An SP22 PVC Screen Plug (38968) is installed in the leading end of the slotted pipe prior to use. The slotted pipe may be cut and the screen plug installed to provide custom screen lengths.

An O-ring is located at the top of each stainless screen and on the screen adapters. When a screen is deployed, this O-ring maintains a seal between the top of the screen and the inner wall of the probe rods or expendable point holder as indicated in Figure 2.1. As a result, any liquid entering the tool string must first pass through the screen.

Screens are constructed such that equipment can be inserted into the screen cavity for sample collection as noted in the following section and illustrated in Figure 2.1, Step 4. This makes direct sampling possible from anywhere within the saturated zone.

The inner rod string and screen are generally removed prior to grouting through the 2.25-inch rod string as shown in Figure 2.1, Steps 5-6. However, a removable plug in the lower end of the screens allows for grouting through flexible tubing extending out the bottom of the screen as with the Geoprobe® SP15/16 Groundwater Samplers if desired.

Sample Collection

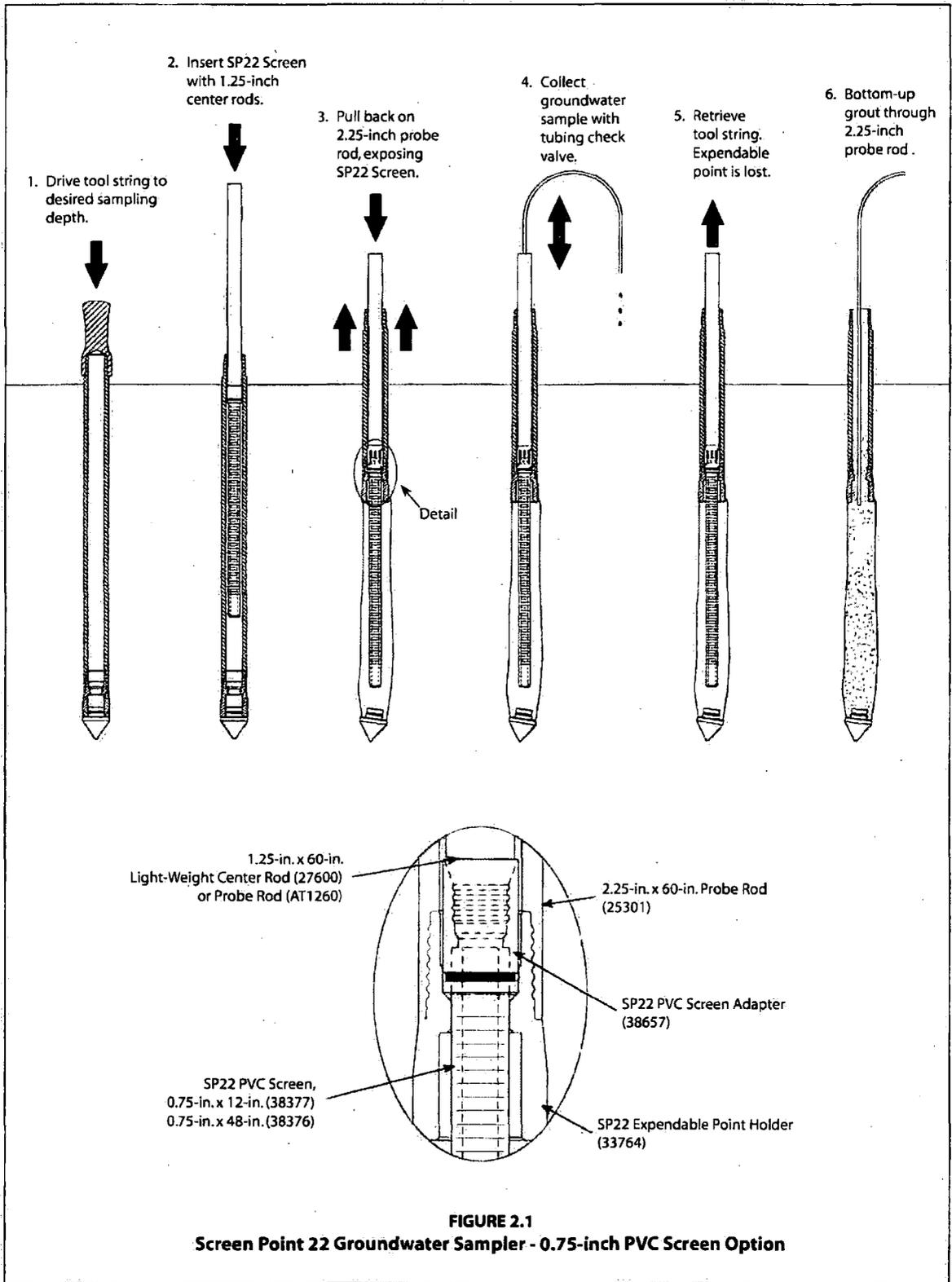
Groundwater samples can be obtained from the SP22 screen in a number of ways. A common method utilizes 0.375-inch OD polyethylene (TB25L) or Teflon® (TB25T) tubing and a check valve assembly. The check valve (with check ball) is attached to one end of the tubing and inserted down the casing until it is immersed in groundwater. Water is then pumped through the tubing and to the ground surface by oscillating the tubing up and down.

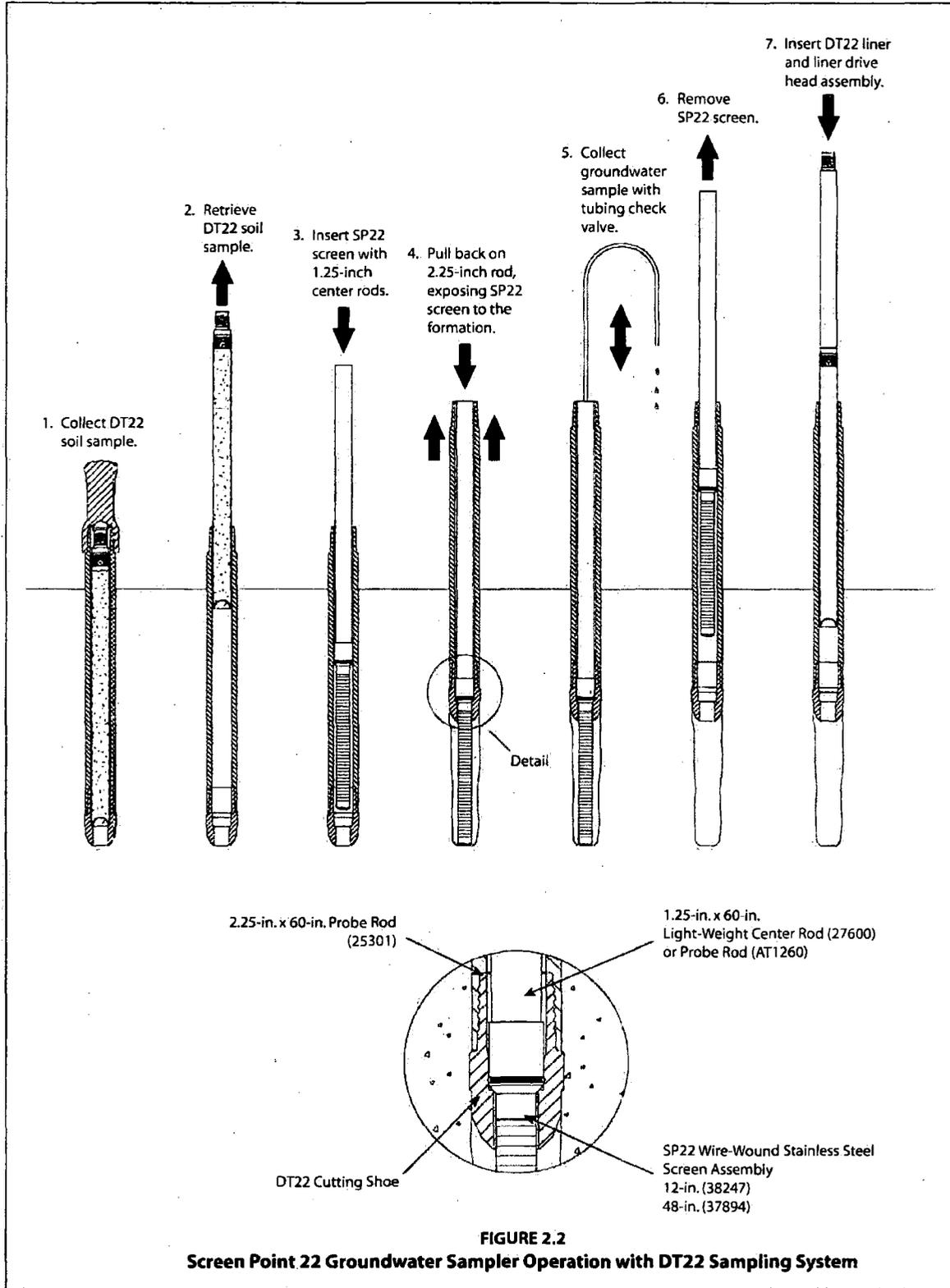
An SP22 Check Valve Assembly (37893) is recommended if sampling through 1.25-inch light-weight center rods. The SP22 Check Valve Assembly is approximately 20 inches long to enable it to pass through the stepped diameters at each rod joint that may cause problems for other, shorter check valves.

An alternative means of collecting groundwater samples is to attach a peristaltic or vacuum pump to tubing that is inserted through the inner rods to within the SP22 screen. This method is limited in that water can be pumped to the surface from a maximum depth of approximately 26 feet (8 m). Another technique for groundwater sampling is to use a stainless steel Mini-Bailer Assembly (GW41). The mini-bailer is lowered down the inside of the casing below the water level where it fills with water and is then retrieved from the casing.

The latest option for collecting groundwater from the SP22 Sampler is to utilize a Geoprobe® MB470 Series Mechanical Bladder Pump (MBP)*. The MBP may be used to meet requirements of the low-flow sampling protocol (Puls and Barcelona 1996, ASTM 2003). Through participation in a U.S. EPA Environmental Technology Verification study, it was confirmed that the MB470 can provide representative samples (EPA 2003).

**The Mechanical Bladder Pump is manufactured under U.S. Patent No. 6,877,965 issued April 12, 2005.*





3.0 TOOLS AND EQUIPMENT

The following tools and equipment can be used to successfully recover representative groundwater samples with the Geoprobe® Screen Point 22 Groundwater Sampler. Refer to Figures 3.1 and 3.2 for identification of the specified parts. Tools are listed below for the most common SP22 / 2.25-inch probe rod configurations. Additional rod sizes and accessories are available. Contact Geoprobe Systems® for information regarding tools and equipment options.

SP22 Sampler Parts	Part Number
SP22 Screen, Wire-Wound Stainless Steel, 4-Slot (48-in.)	37894
SP22 Screen, Wire-Wound Stainless Steel, 4-Slot (12-in.)	38247
Grout Plugs, PE (Pkg. of 25)	GW1552K
SP22 Screen, PVC, 10-Slot, 0.75-in. x 48-in.	38376
<i>SP22 Screen, PVC, 10-Slot, 0.75-in. x 48-inch, Kit (includes 2 each of 38376 and 38429)</i>	38664
SP22 Screen, PVC, 10-Slot, 0.75-in. x 12-in.	38377
<i>SP22 Screen, PVC, 10-Slot 0.75-in. x 12-in., Kit (includes 2 each of 38377 and 38429)</i>	38667
SP22 PVC Screen Plug	38968
<i>SP22 PVC Screen Plug Kit (includes 10 of 38968)</i>	38530
SP22 PVC Screen Adapter, 0.75-in. PVC x 1.25-in. Probe Rod Box	38657
SP22 PVC Screen Head Adapter, 0.75-in. (for flush-threaded 0.75-in. Schedule 40 PVC)	37871
SP22 O-ring Kit (Pkg. of 10 O-rings for SP22 PVC screen adapters and stainless steel screens) ...	37853
O-rings, 0.75-in. PVC Riser (Pkg. of 25)	GW4401R
SP22 Expendable Point Holder, 2.25-in. Probe Rods, AT2045K and 19442 Points	33764
SP22 Expendable Point Holder, 2.25-in. Probe Rods, GW1555 Points*	43128
Outer Casing (2.125-inch Probe Rods) and Inner Rod String	Part Number
Probe Rod, 2.25-in. x 60-in.	25301
Expendable Drive Points, Steel, 2.45-in. OD (Pkg. of 25)	AT2015K
Expendable Drive Points, Steel, 2.45-in. OD, extended shank	19442
Expendable Points, steel, 1.625-in. OD (Pkg. of 25)*	GW1555K
Drive Cap, 2.25-in. Probe Rods, Threadless, (for GH60 and GH80 Series Hammers)	31530
O-Rings, 2.25-in. Probe Rods (Pkg. of 25)	AT2100R
Rod Grip Handle, 2.25-in. Probe Rods, (for GH60 and GH80 Series Hammers)	29385
Light-Weight Center Rod, 1.25-in. x 60-in.	27600
Probe Rod, 1.25-in. x 60-in.	AT1260
O-ring, 1.25-in. rods (Pkg. of 25)	AT1250R
Rod Grip Handle, 1.25/1.5-in. Rods, (for GH60 and GH80 Series Hammers)	15554
PVC Riser, 0.75-in. Schedule 40 x 60-inch	11747
PVC Pipe, 0.75-in. Schedule 40 x 60-inch, 10-Slot	17474
Grout Accessories	Part Number
High-Pressure Nylon Tubing, 0.375-in. OD / 0.25-in. ID, 100-ft. (30 m)	11633
Grout Machine, Auxiliary-Powered	GS2200
Grout System Accessories Package, 2.25-in. rods	GS1015
Groundwater Purging and Sampling Accessories	Part Number
Polyethylene Tubing, 0.375-in. OD, 500 ft.	TB25L
Check Valve Assembly, 0.375-in. OD Tubing x 20 in. Long	37893
Water Level Meter, 0.438-in. OD Probe, 100 ft. cable	GW2000
Mechanical Bladder Pump**	MB470
Mini Bailer Assembly, Stainless Steel	GW41

* Not for use with GH80 Series Hydraulic Hammers

** Refer to the Standard Operating Procedure (SOP) for the Mechanical Bladder Pump (Technical Bulletin No. MK3013) for additional tooling needs.

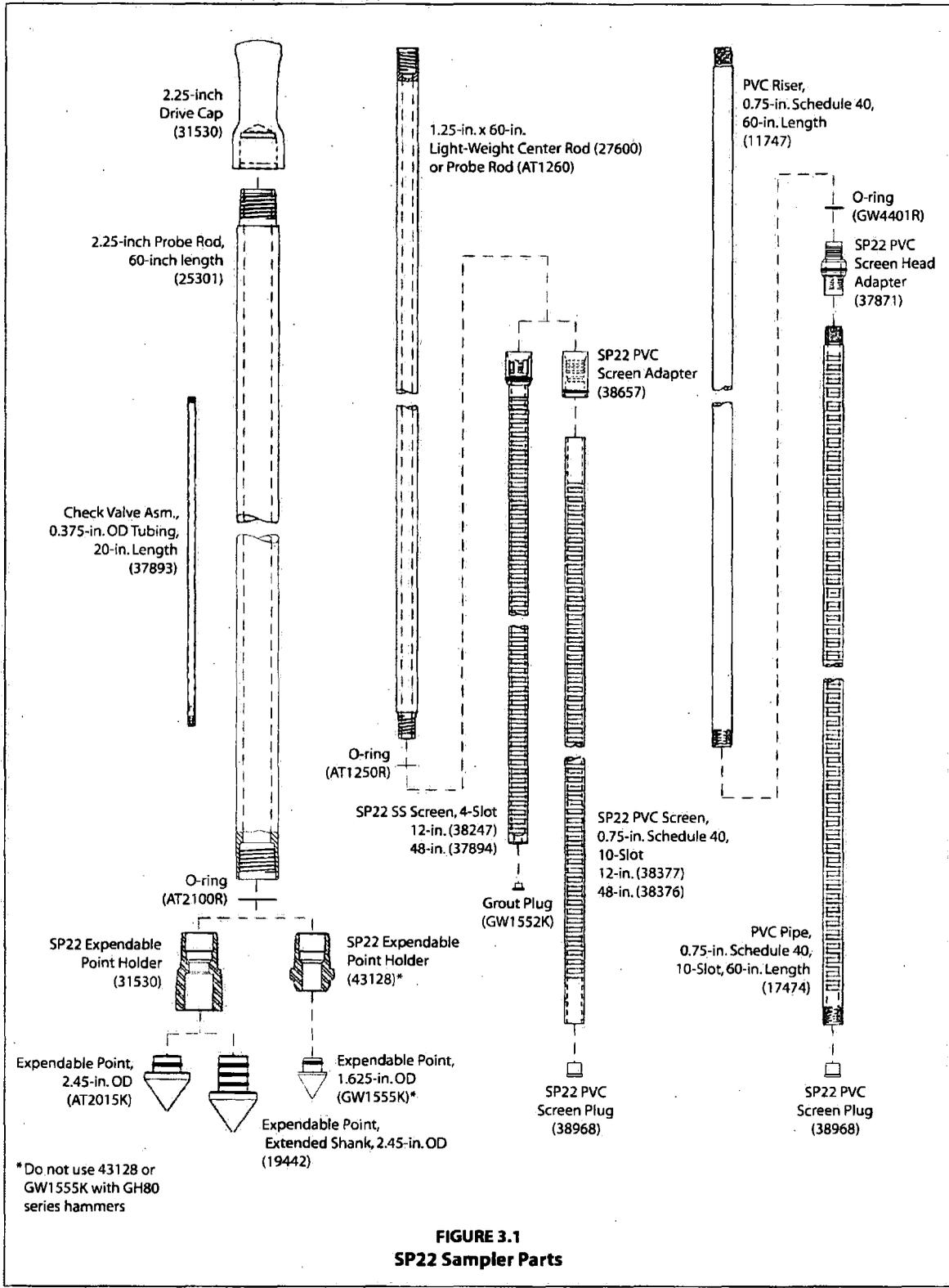


FIGURE 3.1
SP22 Sampler Parts

4.0 OPERATION

4.1 Basic Operation

The SP22 Sampler utilizes a stainless steel or PVC screen which is lowered (post-run) through an alloy steel 2.25-inch OD probe rod tool string. An expendable drive point is placed in an expendable point holder on the leading 2.25-inch probe rod prior to advancement (Fig. 4.1). This expendable point holder is removed and stays in the subsurface as the rods are pulled back to exposes the SP22 screen. O-rings on the probe rods, the expendable point holder, and the expendable drive point provide a watertight tool string which keeps contaminants out of the system as the 2.25-inch rods are driven to depth in preparation for installation of the SP22 screen.

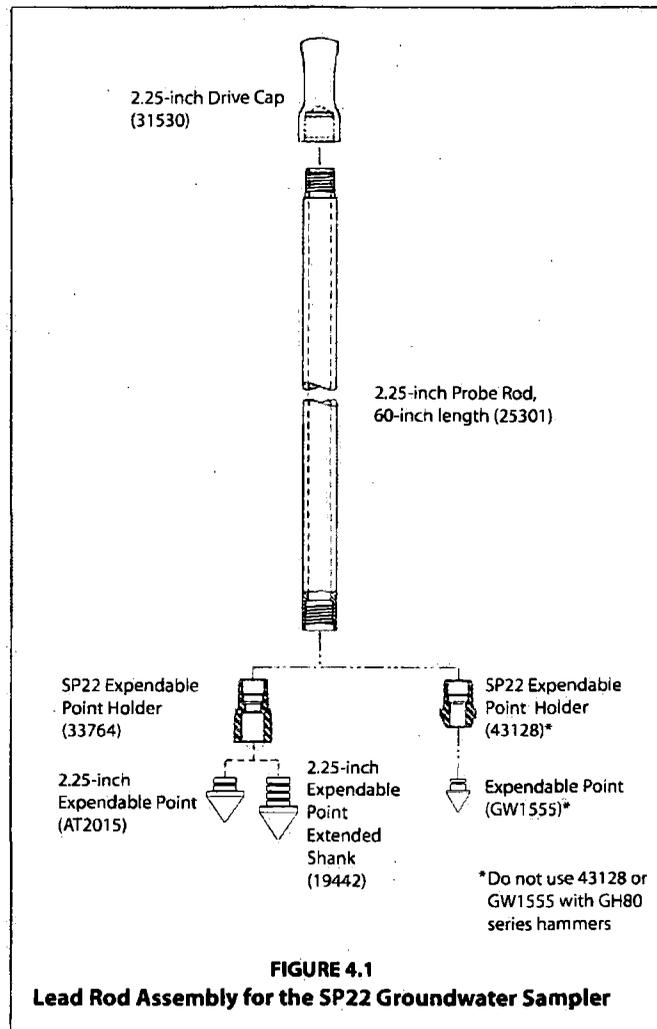
Once the sampling interval is reached with the 2.25-inch probe rods, the stainless steel or PVC screen is lowered through the rods using 1.25-inch probe rods, 1.25-inch light-weight center rods, or 0.75-inch PVC riser pipe. The 2.25-inch tool string is then retracted while the screen is held in place with the inner rods or riser. The system is now ready for groundwater sampling. When sampling is complete, the inner rods and screen are removed for grouting during retrieval or the 2.25-inch rods. Alternatively, a removable plug is located in the bottom of the screens to allow grouting directly through the inner tool string with high-pressure tubing during retrieval.

4.2 Decontamination

In order to collect representative groundwater samples, all sampler parts must be thoroughly cleaned before and after each use. Scrub all metal parts using a stiff brush and a nonphosphate soap solution. Steam cleaning may be substituted for hand-washing if available. Rinse with distilled water and allow to air-dry before assembly.

4.3 Lead Rod Assembly (Fig. 4.1)

1. Place an O-ring on the expendable point holder.
2. Thread expendable point holder into the 2.25-inch probe rod.
3. Place an O-ring on a steel expendable drive point.
4. Firmly seat the expendable point in the expendable point holder.
5. Place 2.25-inch Drive Cap (31530) on the top of the 2.25-inch probe rod. The lead rod assembly is now ready to be driven to depth.



4.4 Advancing the Tool String (Fig. 4.2, step 1)

To provide adequate room for screen deployment with the Rod Grip Pull System, the probe derrick should be extended a little over halfway out of the carrier vehicle when positioning for operation.

1. Drive first 2.25-inch probe rod (as assembled in section 4.3).
2. Advance the tool string at a slow speed for the first few feet to ensure that the string is aligned properly.
3. Completely raise the hammer assembly. Remove the drive cap and place an O-ring in the top groove of the driven probe rod. Distilled water may be used to lubricate the O-ring if needed.

Add a probe rod (length to be determined by operator) and reattach the drive cap to the rod string. Drive the tool string the entire length of the new rod.

4. Repeat Step 3 until the desired sampling interval is reached. Approximately 12 inches (305 mm) of the last probe rod must extend above the ground surface to allow attachment of the puller assembly. A 12-inch (305 mm) rod may be added if the tool string is over-driven.
5. Remove the drive cap and retract the probe derrick away from the tool string.

4.5 Screen Deployment (Fig 4.2, step 2 - 4)

1. Attach an SP22 stainless steel or PVC screen to a 1.25-inch probe rod, 1.25-inch light-weight center rod, or 0.75-inch flush-thread PVC riser using an SP22 PVC Screen Adapter (38657) or SP22 PVC Screen Head Adapter (37871) as shown in Figure 3.1. Note that the 38657 screen adapter is connected to the SP22 PVC screen using the setscrews provided with the adapter.

and lower it into the driven casing.

2. Lower the screen into the 2.25-inch probe rod casing and add rods or riser until the screen head contacts the bottom of the tool string.
3. Ensure that at least 48 inches (1219 mm) of rods or riser protrudes from the top 2.25-inch probe rod.
4. Maneuver the probe assembly into position for pulling.
5. Raise (pull) the outer 2.25-inch tool string while physically holding the screen in place with the inner 1.25-inch rods or 0.75-inch riser. A slight knock with the inner tool string will help to dislodge the expendable point and start the screen moving inside the probe rod.

Raise the hammer and outer tool string to expose the desired length of screen. The inner rods will begin raising with the outer rods when the screen adapter contacts the necked portion of the expendable point holder or DT22 Cutting Shoe. Use care when deploying a PVC screen so as not to break the screen when it contacts the expendable point.

6. Remove the rod grip handle, lower the hammer assembly, and retract the probe derrick. Remove the top 2.25-inch probe rod.
7. Groundwater samples can now be collected with a mini-bailer, peristaltic or vacuum pump, tubing bottom check valve assembly, bladder pump, or other acceptable small diameter sampling device.

When inserting tubing or a bladder pump down the rod string, ensure that it enters the screen interval. The leading end of the tubing or bladder pump will sometimes catch at the screen head giving the illusion that the bottom of the screen has been reached. An up-and-down motion combined with rotation helps move the tubing or bladder pump past the lip and into the screen.

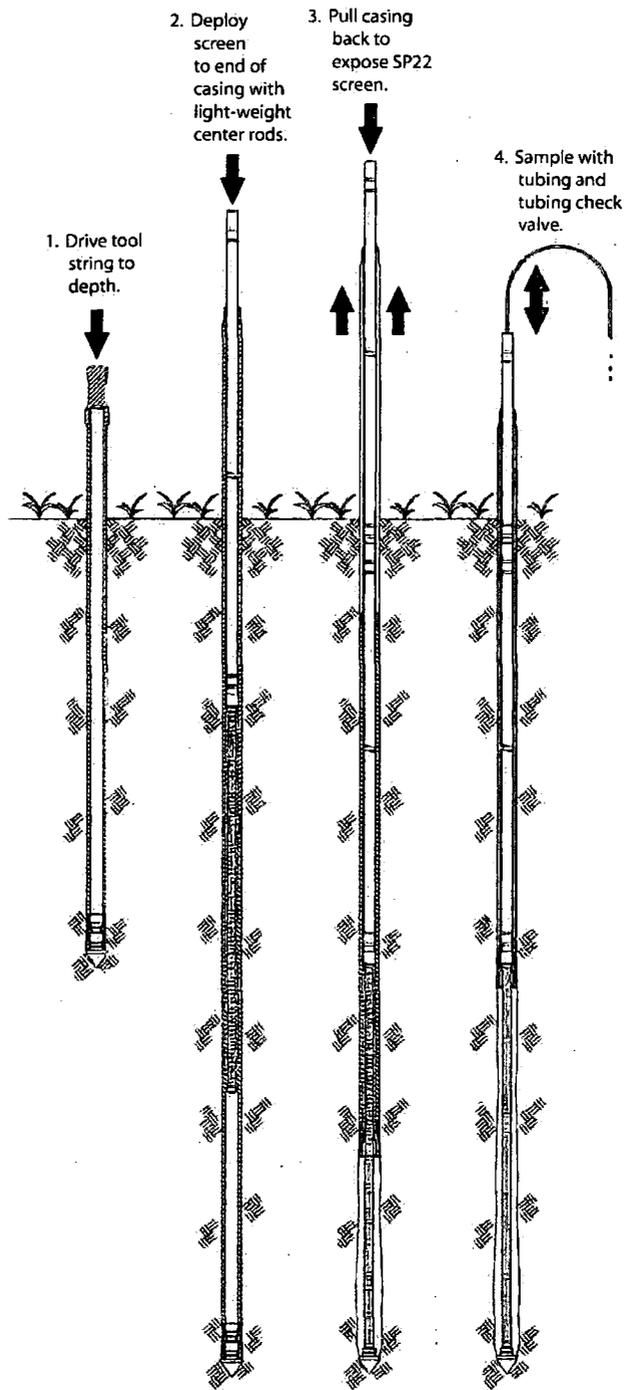


FIGURE 4.2
Screen Deployment for SP22 Sampler

4.6 Abandonment Grouting for SP22 Screens

The SP22 Sampler can meet ASTM D 5299 requirements for abandoning environmental wells or borings when grouting is conducted properly. A removable grout plug makes it possible to deploy tubing through the bottom of the SP22 screens, but the easiest method is to remove the inner string of rods, including the SP22 screen. A Grout Machine is then used to pump grout into the open probe hole as the outer casing is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations. (Figure 4.3)

1. Maneuver the probe assembly into position for pulling.
2. High-Pressure Nylon Tubing (11633) is inserted down through the probe rods through the bottom of the expendable point holder (Fig. 4.3).

Note: All probe rods remain strung on the tubing as the tool string is pulled. Provide extra tubing length to allow sufficient room to lay the rods on the ground as they are removed. An additional 20 feet is generally enough.

3. Operate the grout pump while pulling the first rod with the rod grip pull system. Coordinate pumping and pulling rates so that grout fills the void left by the sampler. After pulling the first rod, release the rod grip handle, fully lower the hammer, and regrip the tool string. Unthread the top probe and slide it over the tubing placing it on the ground near the end of the tubing.
4. Repeat Step 5 until the tool string is retrieved. Do not bend or kink the tubing when pulling and laying out the probe rods. Sharp bends create weak spots in the tubing which may burst when pumping grout. Remember to operate the grout pump only when pulling the rod string. The probe hole is thus filled with grout from the bottom up as the rods are extracted.
5. Promptly clean all probe rods and sampler parts before the grout sets up and clogs the equipment.

4.7 Retrieving the Screen Point 22 Sampler

If grouting is not required, the Screen Point 22 Sampler can be retrieved by pulling the probe rods as with most other Geoprobe® applications. The Rod Grip Pull System should be used for this process as it allows the operator to remove rods without completely releasing the tool string. This avoids having the probe rods fall back downhole when released during the pulling procedure. A standard Pull Cap (33622) may still be used if preferred. Refer to the Owner's Manual for your Geoprobe® direct push machine for specific instructions on pulling the tool string.

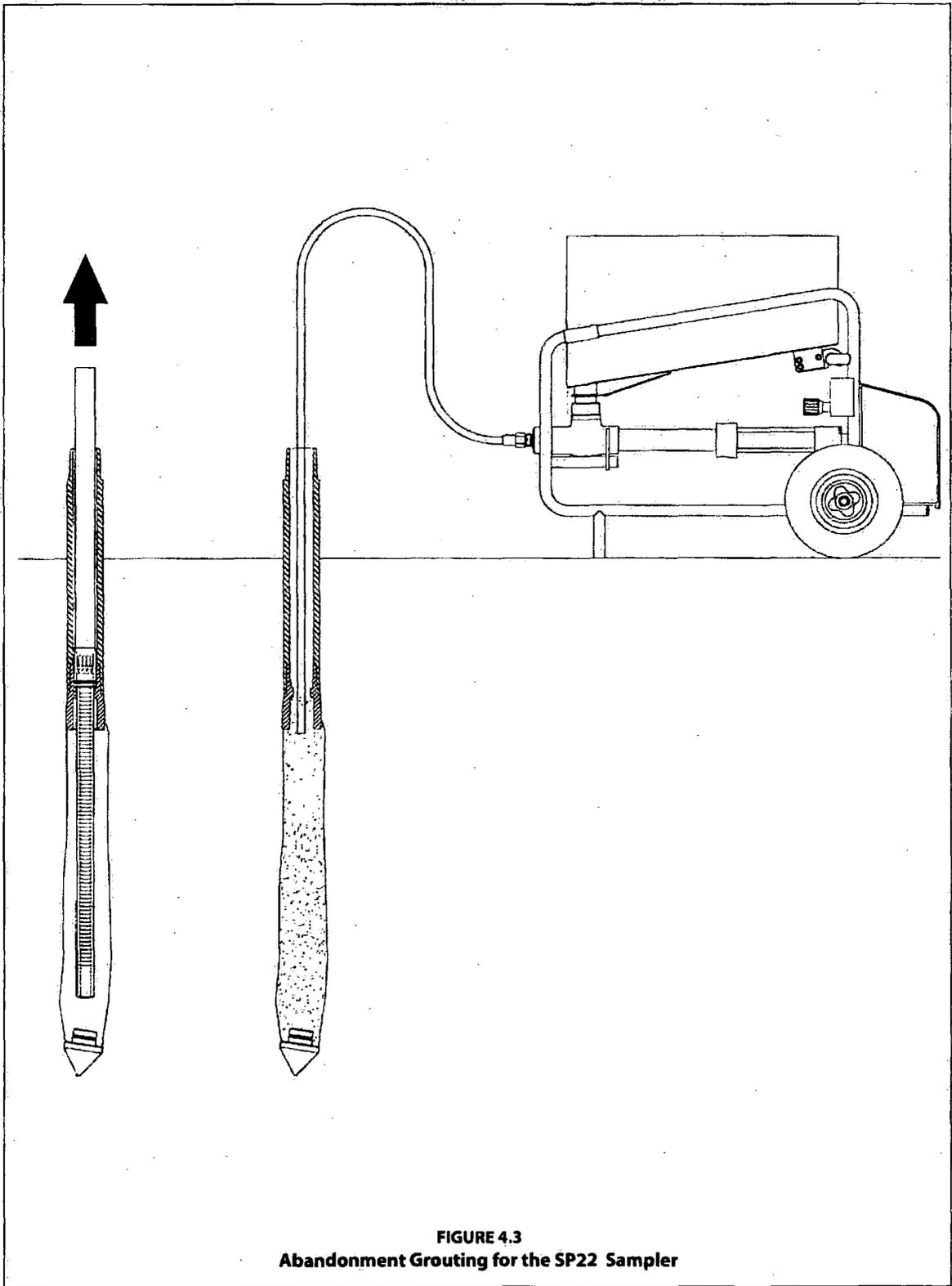


FIGURE 4.3
Abandonment Grouting for the SP22 Sampler

5.0 REFERENCES

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