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SAMPLING AND ANALYSIS PLAN FOR OPERABLE UNIT 2 (OU 2) PRE DESIGN INVESTIGATION NSY PORTSMOUTH ME

11/1/2010

TETRA TECH NUS

Sampling and Analysis Plan for Operable Unit 2 Pre-Design Investigation

Portsmouth Naval Shipyard

Kittery, Maine

Naval Facilities Engineering Command Mid-Atlantic

Contract Number N62470-08-D-1001
Contract Task Order WE43

November 2010

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

SAMPLING AND ANALYSIS PLAN FOR
OPERABLE UNIT 2 PRE-DESIGN INVESTIGATION

PORTSMOUTH NAVAL SHIPYARD
KITTERY, MAINE

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

Prepared by:
Tetra Tech, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, Pennsylvania 19406

Prepared under:
Contract Number N62470-08-D-1001
Contract Task Order WE43

NOVEMBER 2010

Tetra Tech Approval Signatures:

Daniel C Witt 11-10-10

Daniel Witt, P.E.
Project Manager
Tetra Tech, Inc.

T. Johnston 11-10-10

Tom Johnston, Ph.D.
QA Manager
Tetra Tech, Inc.

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

SAMPLING AND ANALYSIS PLAN FOR
OPERABLE UNIT 2 PRE-DESIGN INVESTIGATION

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NOVEMBER 2010

Approval Signatures :

Matthew Audet
Regulatory Remedial Project Manager
USEPA Region 1

Iver McLeod
Remedial Project Manager
Maine Dept. of Environmental Protection

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

Draft

**SAMPLING AND ANALYSIS PLAN FOR
OPERABLE UNIT 2 PRE-DESIGN INVESTIGATION**

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Contract Task Order WE43**

JUNE 2010

Approval Signatures :

L. L. Cole 6/23/2010 Kenneth Bowers 6/21/10

L. L. Cole, P.E.
Remedial Project Manager
NAVFAC MIDLANT
Northeast IPT
Code OPTE3-2

Kenneth Bowers
NAVFAC QA Officer
NAVFAC LANT

SAP Worksheet #1D – Maine Certified Geologist Approval Page

(UFP-QAPP Manual Section 2.1)

By affixing my seal to the "*Sampling and Analysis Plan for Operable Unit 2 Pre-Design Investigation, Portsmouth Naval Shipyard, Kittery, Maine,*" prepared by Tetra Tech, Inc., and dated (November 2010), I am certifying that the geologic information and interpretations herein are true and correct to the best of my knowledge. I further certify that I am licensed to practice in the State of Maine and that it is within my professional expertise to verify the correctness of the information.

STATE OF MAINE
TIMOTHY S.
EVANS
GE505
GEOLOGIST

EXECUTIVE SUMMARY

This Sampling and Analysis Plan (SAP) for the Operable Unit (OU2) Pre-Design Investigation provides the basis and methods for completing a Pre-Design Investigation at OU2 at Portsmouth Naval Shipyard (PNS), Kittery, Maine. Information necessary to complete the remedial action for OU2 will be collected as part of this investigation. This SAP follows the format of the Uniform Federal Policy (UFP) for Quality Assurance Project Plans (QAPPs) (USEPA, March 2005), and was prepared to address a one-time investigation at OU2.

Tetra Tech, Inc. (Tt) prepared this UFP-SAP for the United States Department of Navy, Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract Number N62470-08-D-1001, Contract Task Order (CTO) WE43. This investigation is being conducted to support of the OU2 Remedial Design (RD) under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). The information collected during this investigation combined with information from previous investigations is intended to support evaluation of the extent of contamination.

OU2 is located in the south-central portion of PNS and currently consists of Site 6 – The Defense Reutilization and Marketing Office (DRMO) Storage Yard, Site 29 – the Former Teepee Incinerator Site, and the DRMO Impact Area.

Based on background information, previous investigations, the conceptual site model (CSM), and problem statements, it was determined that additional soil data are needed to define the limit of soil contamination on the western side of OU2, in the vicinity of Building 348 and John Paul Jones Avenue, to support remedial decision-making. Based on site history, investigations, and the CSM, lead, copper, and polychlorinated biphenyls (PCBs) are the contaminants of concern (COCs) intended to be delineated by this study. Additional information required to complete the RD include a wetlands functional value and habitat assessment of the intertidal zone and topographic mapping of the site. The wetland functions and values assessment will document the current conditions and serve as a baseline to determine if there are changes in wetland functions and values in the future. The topographic survey will be used to revise the existing conditions base map for the RD drawings.

The OU2 sampling strategy consists of collecting soil samples at locations spaced approximately 25 feet apart across the study area, plus of two additional locations, to provide data to delineate remediation areas. Nineteen soil borings, with soil samples planned for collection at 2-foot intervals, will be competed to approximately 10 feet below ground surface (bgs), which generally corresponds to the top of the tidally saturated zone in this area. The 0- to 10-foot-bgs depth was used to span the zone for which human

exposure was assumed in the Supplemental Remedial Investigation (RI) Report to evaluate human health risks.

The soil sample analyses will be conducted in phases based on the analytical results of the surface (0- to 2-foot-bgs) soil samples. If the results of the surface and shallow subsurface soil samples sent for analysis are greater than the project action levels, the next phase (i.e., depth interval) of samples will be analyzed. Expedited laboratory turnaround times will be employed so that decisions can be made concerning whether to analyze the next depth interval of samples while still maintaining holding times.

A report of the results will be prepared, and the OU2 RD will be completed at the conclusion of this investigation.

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Figure 5: Proposed Sampling Locations

Appendices

Appendix A Field Documentation Forms
Appendix B Field Standard Operating Procedures
Appendix C Laboratory Standard Operating Procedures and Certifications
Appendix D Meeting Minutes

ACRONYMS AND ABBREVIATIONS

AES	atomic emission spectrometer
AOC	area of concern
bgs	below ground surface
CA	Corrective action
CAS	Chemical Abstract Service
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CG	Certified Geologist
CLEAN	Comprehensive Long-Term Environmental Action Navy
COC	chemical of concern
CSM	conceptual site model
CTO	Contract Task Order
%D	percent difference
DFTPP	decafluorotriphenylphosphine
DoD	Department of Defense
DPT	Direct-push technology
DQI	data quality indicator
DQO	data quality objective
DRMO	Defense Reutilization and Marketing Office
DVM	Data Validation Manager
EDD	Electronic Data Deliverable
EERA	Estuarine Ecological Risk Assessment
ELAP	Environmental Laboratory Accreditation Program
FCS	Final Confirmation Study
FOL	Field Operations Leader
FS	Feasibility Study
FTMR	Field Task Modification Request
FWENC	Foster Wheeler Environmental Corporation
GC	Gas Chromatograph
MS	Mass Spectrometer
GPS	Global Positioning System
HASP	Health and Safety Plan
HI	Hazard Index
HSM	Health and Safety Manager

ICP	Inductively Coupled Plasma
ICS	Interference Check Standards
ICV	Initial Calibration Verification
IDW	Investigation-derived waste
IR	Installation Restoration
IS	Internal Standard
LANT	Atlantic
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
LUC	Land Use Control
MDL	Method Detection Limit
MEDEP	Maine Department of Environmental Protection
MPC	Measurement Performance Criterion
MS/MSD	Matrix spike/matrix spike duplicate
NAVFAC	Naval Facilities Engineering Command
NCCOSC	Naval Center Combat and Operational Stress Control
NFESC	Naval Facilities Engineering Service Center
NIRIS	Naval Installation Restoration Information Solution
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PAH	polycyclic aromatic hydrocarbon
PAL	Project Action Limit
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
PCB	polychlorinated biphenyl
PID	Photoionization detector
PM	Project Manager
PNS	Portsmouth Naval Shipyard
POC	Point of Contact
PRG	Preliminary Remediation Goal
PWD	Public Works Department
QA	quality assurance
QAM	Quality Assurance Management
QAPP	Quality Assurance Project Plan
QC	quality control
QL	Quantitation Limit

QSM	Quality Systems Manual
%R	Percent Recovery
RAB	Restoration Advisory Board
RCRA	Resource Conservation and Recovery Act
RD	remedial design
RFI	RCRA Facility Investigation
RI	Remedial Investigation
RPD	Relative Percent Difference
RRT	Relative Retention Time
%RSD	percent relative standard deviation
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SD	Serial Dilution
SHSC	Site Health and Safety Coordinator
SIM	Selective Ion Monitoring
SOP	Standard Operating Procedure
SQL	Structured Query Language
SSO	Site Safety Officer
TBD	To be determined
Tt	Tetra Tech, Inc.
UFP	Uniform Federal Policy
USEPA	United States Environmental Protection Agency
WDA	Waste Disposal Area

SAP Worksheet #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: **Site 6 and Site 29**
OU: **2**
Contractor Name: **Tt**
Contract Number: **N62470-08-D-1001**
Contract Title: **CLEAN**
Work Assignment Number: **CTO WE43**

1. This OU2 Pre-Design Investigation SAP was prepared per guidelines in the United States Environmental Protection Agency's (U.S. EPA) UFP for QAPPs (March 2005) and Guidance for Quality Assurance Project Plans, (U.S. EPA, December 2002).

2. Identify regulatory program: CERCLA

3. This document is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Internal Meeting	July 9 and 13, 2009
Meeting with Regulators	December 8, 2009
Follow-Up Conference Call with Regulators	March 23, 2010

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

Title	Date	ARF Number
OU2 Additional Investigation QAPP	October 2007	N00102.AR.001626

6. List organizational partners (stakeholders):

USEPA Region 1
Maine Department of Environmental Protection (MEDEP)
Natural Resource Trustees
PNS Restoration Advisory Board (RAB)

Lead organization: Navy – NAVFAC Mid-Atlantic

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

Not applicable

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	Not Applicable
2	Table of Contents SAP Identifying Information	Not Applicable
3	Distribution List	Not Applicable
4	Project Personnel Sign-Off Sheet	Not Applicable
Project Organization		
5	Project Organizational Chart	Not Applicable
6	Communication Pathways	Not Applicable
7	Personnel Responsibilities and Qualifications Table	Not Applicable
8	Special Personnel Training Requirements Table	Not Applicable
Project Planning/ Problem Definition		
9	Project Planning Session Documentation (including Data Needs Tables) Project Scoping Session Participants Sheet	Not Applicable
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	Not Applicable
11	Site-Specific Project Quality Objectives	Not Applicable
12	Measurement Performance Criteria Table	Not Applicable
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	Not Applicable
14	Summary of Project Tasks	Not Applicable
15	Reference Limits and Evaluation Table	Not Applicable
16	Project Schedule/Timeline Table	Not Applicable
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	Not Applicable
18	Sampling Locations and Methods/SOP Requirements Table Sample Location Map(s)	Not Applicable
19	Analytical Methods/SOP Requirements Table	Not Applicable
20	Field Quality Control Sample Summary Table	Not Applicable
21	Project Sampling SOP References Table Sampling SOPs	Not Applicable
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Not Applicable
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	Not Applicable
24	Analytical Instrument Calibration Table	Not Applicable
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Not Applicable

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	Not Applicable
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	Not Applicable
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	Not Applicable
Data Management Tasks		
29	Project Documents and Records Table	Not Applicable
30	Analytical Services Table Analytical and Data Management SOPs	Not Applicable
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	Not Applicable
32	Assessment Findings and Corrective Action Responses Table	Not Applicable
33	QA Management Reports Table	Not Applicable
D. Data Review		
34	Verification (Step I) Process Table	Not Applicable
35	Validation (Steps IIa and IIb) Process Table	Not Applicable
36	Validation (Steps IIa and IIb) Summary Table	Not Applicable
37	Usability Assessment	Not Applicable

SAP Worksheet #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipient	Title/Role	Organization	Telephone Number (Optional)	E-Mail Address
Linda Cole	Navy Remedial Project Manager (RPM)/Manages Project	NAVFAC Mid-Atlantic	(757) 341-2011	linda.cole@navy.mil
Matthew Audet	USEPA RPM/Manages Project	U.S. EPA Region 1	(617) 918-1449	audet.matthew@epa.gov
Iver McLeod	MEDEP RPM/Manages Project	MEDEP	(207) 287-8010	iver.j.mcleod@maine.gov
Matt Thyng	Public Works Department (PWD) Maine Installation Restoration (IR) Program Manager/Shipyard Point of Contact(POC)	NAVFAC Mid-Atlantic	(207) 438-6618	frederick.thyng@navy.mil
Deborah Cohen	Facility Coordinator/Manages Tt work pertaining to the shipyard	Tt	(412) 921-7118	deborah.cohen@tetratech.com

Name of SAP Recipient	Title/Role	Organization	Telephone Number (Optional)	E-Mail Address
Daniel Witt	Project Manager (PM)/Manages project on a daily basis (distributes copies of SAP to others implementing the SAP at Tt)	Tt	(412) 921-8259	daniel.witt@tetratech.com
Tim Evans	Tt Maine Certified Geologist (CG)/Reviews and approves project geological interpretations	Tt	(412) 921-7281	tim.evans@tetratech.com
Edward Lawler	PM/Manages project	Mitkem Laboratories	(401) 732-3400	elawler@mitkem.com

SAP Worksheet #4 -- Project Personnel Sign-Off Sheet

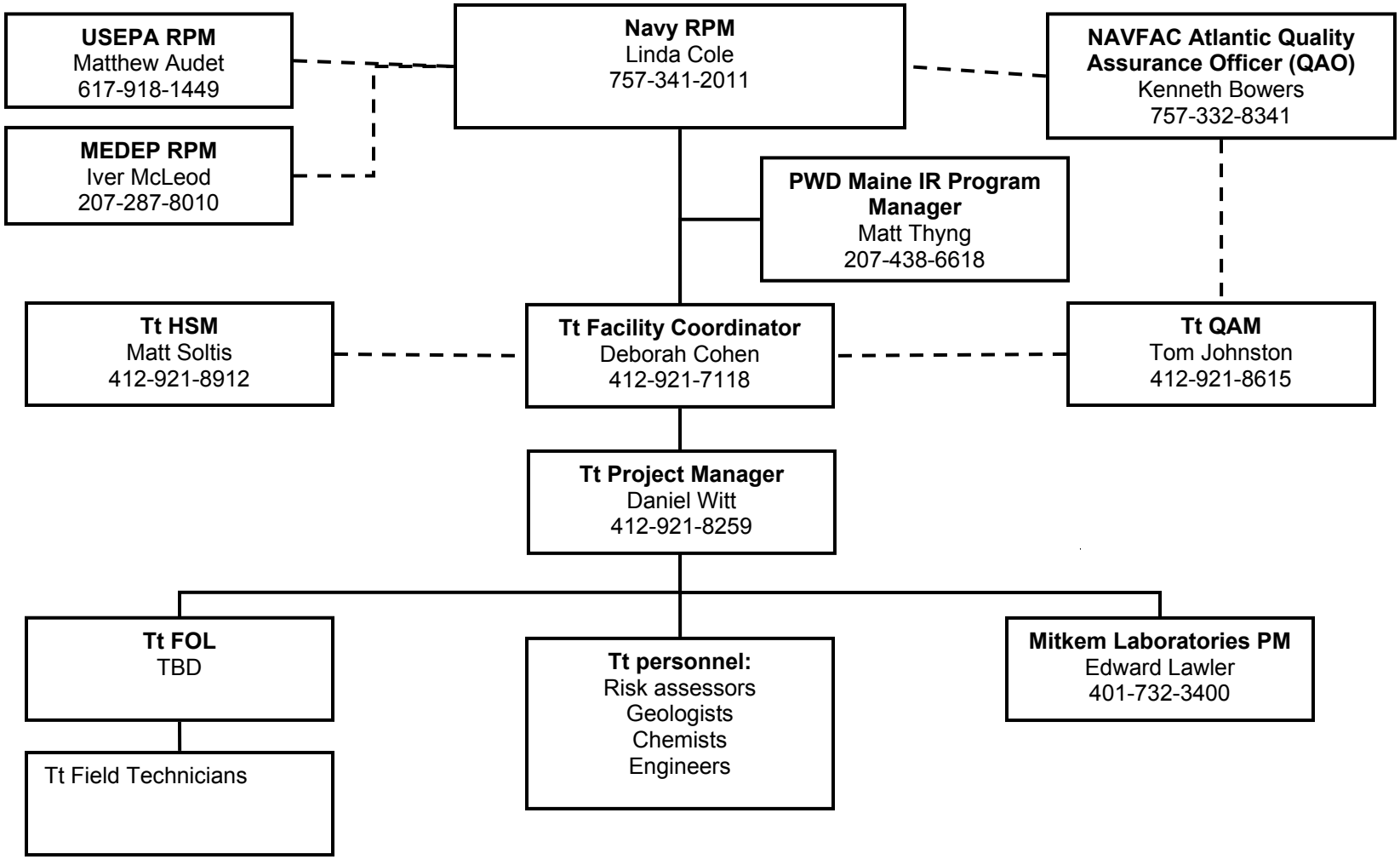
(UFP-QAPP Manual Section 2.3.2)

Name	Organization/Title/Role	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Deborah Cohen	Tt/FacilityCoordinator/ Manages Tt work pertaining to PNS		All	
Daniel Witt	Tt/PM/Manages project	See Worksheet #1A Tt Title and Approval Page	All	
Tom Johnston	Tt/ Quality Assurance Manager (QAM)/Reviews and oversees Tt CLEAN Program QA	See Worksheet #1A Tt Title and Approval Page	All	
Tim Evans	Tt/Maine CG/Reviews and approves all geological interpretations and decisions	See Worksheet #1D Maine CG Approval Page	All	
Joseph Samchuck	Tt/Data Validation Manager (DVM)/Oversees and approves data validation		Worksheet #s 12, 14, 15, 19, 20, 23-28, 30, 34-37	
Matt Soltis	Tt/Health and Safety Manager (HSM)/Reviews and approves site Health and Safety Plan (HASP)		HASP	
TBD	Tt/Field Operations Leader (FOL)/Ensures that field operations are carried out in accordance with this SAP		All	

Name	Organization/Title/Role	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Matthew Kraus	Tt/Project Chemist/ Coordinates with the laboratory and provides project chemistry support		All	
Edward Lawler	Mitkem Laboratories/ PM/Manages project		Worksheet #s 12, 14, 15, 19, 20, 23-28, 30, 34-37	

SAP Worksheet #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)



Contact Linda Cole for project personnel contact information.

SAP Worksheet #6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.1)

Communication Driver	Responsible Affiliation	Name	Phone Number	Procedure
SAP amendments	Tt PM NAVFAC RPM	Daniel Witt Linda Cole	412-921-8259 757-341-2011	The Tt PM will send the NAVFAC RPM a concurrence letter within 7 days of realizing an amendment is needed. The NAVFAC RPM will sign the letter within 5 days of receipt.
Schedule changes	Tt PM	Daniel Witt	412-921-8259	The Tt PM will send NAVFAC a schedule concurrence letter within 7 days of realizing a schedule impact or prior to the first affected deliverable date.
Field issues that require changes in scope or implementation of field work	Tt FOL Tt PM NAVFAC RPM	TBD Daniel Witt Linda Cole	TBD 412-921-8259 757-341-2011	The Tt FOL will inform the Tt PM verbally the day the issue is realized. The Tt PM will inform the NAVFAC RPM of the issue verbally within 1 day of the FOL's notification. The Tt PM will also send a concurrence letter to the NAVFAC RPM within 7 days if project scope is affected. The NAVFAC RPM will sign the letter within 5 days of receipt. Project personnel will document changes on a Field Task Modification Request (FTMR) form. The Tt PM will place the form in the project file, with approval signatures as determined by the Tt PM.

Communication Driver	Responsible Affiliation	Name	Phone Number	Procedure
Stop work recommendations, for example, to protect workers from unsafe conditions/situations or to prevent a degradation in quality of work	Tt FOL/Site Safety Officer (SSO) Tt PM Tt QAM	TBD Daniel Witt Tom Johnston	TBD 412-921-8259 412-921-8615	On the day the problem is identified, the Tt FOL, Tt SSO, or Tt QAM will inform the Tt PM verbally and document the problem on an FTMR. On the same day of the notification from the Tt FOL, the Tt PM will inform the NAVFAC and Installation RPMs via e-mail.
Chemical data issues	Laboratory PM Tt Project Chemist	Ed Lawler Matt Kraus	401-932-3400 412-921-8729	When issue is related to chemical data, the Laboratory PM will notify the Tt Project Chemist verbally within 2 days of identification of problem. The Project Chemist will notify the Tt PM verbally within 1 day.

SAP Worksheet #7 – Personnel Responsibilities and Qualifications Table

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Linda Cole	RPM/Manages project	NAVFAC Mid-Atlantic	Oversees project implementation, including scoping, data review, and evaluation.
Iver McLeod	RPM/Manages Project	MEDEP	Participates in scoping, data review and evaluation, and SAP preparation.
Matthew Thyng	PWD Maine IR Program Manager/Manages daily site activities related to this project	PNS	Oversees site activities, participates in scoping, data review and evaluation, and reviews the SAP.
Matthew Audet	RPM/Manages project	U.S. EPA Region 1	Participates in scoping, data review and evaluation, and SAP preparation.
Deborah Cohen	Facility Coordinator/Manages Tt work pertaining to PNS	Tt	Oversees and manages Tt responsibilities associated with the shipyard.
Daniel Witt	PM/Manages project on a daily basis	Tt	Oversees project, financial, schedule, and technical day-to-day management of the project.
TBD	FOL/Manages field operations	Tt	Supervises, coordinates, and performs field sampling activities.
TBD	SSO/Oversees site activities to ensure that safety requirements are met	Tt	Responsible for on-site project-specific health and safety training and monitoring site conditions. Details of these responsibilities are presented in the HASP.
Tom Johnston	QAM/Oversees program and project QA activities	Tt	Ensures that the quality aspects of the CLEAN program are implemented, documented, and maintained.
Joseph Samchuck	DVM/Oversees data validation activities	Tt	Manages data validation activities within Tt, including: • Ensuring QA of data validation deliverables. • Providing technical advice on data usability. • Coordinating and maintaining data validation review schedule.
Matt Soltis	HSM/Oversees health and safety activities	Tt	Oversees the Tt CLEAN Health and Safety Program.
Matthew Kraus	Project Chemist/Oversees review of data quality and coordinates with the laboratory	Tt	Participates in project scoping, prepares laboratory scopes of work, and coordinates laboratory-related functions with laboratory. Oversees data quality reviews and QA of data validation deliverables.
Edward Lawler	Laboratory PM/Manages project	Mitkem Laboratories	Coordinates analyses with laboratory chemists, ensures that the scope of work is followed, provides QA of data packages, and communicates with Tt project staff.

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker performing sampling of hazardous materials will be required to have completed a 40-hour course (and annual 8-hour refresher) in health and safety training as described under Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120(b)(4). Safety requirements are addressed in greater detail in the Tt site-specific HASP.

Mitkem Laboratories, the selected analytical laboratory, is accredited under the Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP). The DoD ELAP Certification for Mitkem Laboratories is in Appendix C.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name: PNS OU2 Pre-Design Investigation **PM**: Daniel Witt	**Site Name**: OU2 **Site Location**: PNS, Kittery, Maine

Date of Session: July 9, 2009 and July 13, 2009
Scoping Session Purpose: Tt internal discussion to determine draft DQOs

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
Anna-Marie Christian	Statistician	Tt	Contact Linda Cole	Contact Linda Cole	Statistician
Deborah Cohen, P.E.	Process Engineer	Tt	Contact Linda Cole	Contact Linda Cole	PNS Facility Coordinator/Engineer
Ronald Kotun, Ph.D.	Senior Toxicologist	Tt	Contact Linda Cole	Contact Linda Cole	DQO Facilitator and Risk Assessor
Daniel Witt, P.E.	PM/Civil Engineer	Tt	Contact Linda Cole	Contact Linda Cole	PM/Engineer

Comments/Decisions:

- The problem definition was discussed and it was decided that the problem definition would include the collection of additional data to define the limit of contamination on the western side of OU2 to complete the RD.

- The decision statement was discussed and it was decided to include the following: "If the soil contaminant concentrations obtained during this pre-design investigation are less than or equal to the residential PRGs presented in the OU2 Feasibility Study, then the limit of contamination presented in the Feasibility Study will not change. If the contaminant concentrations are greater than the OU2 residential PRGs, the limit of contamination will be refined for the remedial design."

- It was agreed that Project Action Levels (PALs) would only include residential PRGs because the project decision is limited to deciding if an area would be included as part of OU2.

- The decision was made that data will be collected on 30-foot centers across the study boundary and at 2-foot depth intervals until encountering refusal (note: through later development of this plan, the spacing of soil borings was refined to be approximately 25 feet apart). All samples will be analyzed for contaminants of concern (COCs) by a fixed-base laboratory. If the performance criteria indicated that additional samples are required, then the sample spacing will be reconsidered.

Action Items:

- The statistical elements of the performance criteria will be developed to ensure that sufficient samples are collected to characterize contamination in the study area.

- The internal draft version of Worksheet #s 10 and 11 will be revised based on these discussions and sent to the Navy to review on July 15, 2009.

Consensus Decisions:

See Comments/Decisions above.

Project Name: OU2 Pre-Design Investigation Projected Date(s) of Sampling: November 2010 **PM**: Daniel Witt	**Site Name**: Operable Unit 2 **Site Location**: DRMO Area Western End

Date of Session: December 8, 2009, with a follow-up call on March 23, 2010
Scoping Session Purpose: Discuss Draft DQOs

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Linda Cole	RPM	NAVFAC MIDLANT	757-341-2011	linda.cole@navy.mil	RPM
Matt Thyng (1)	IR Program Manager	PWD-ME	Contact Linda Cole	Contact Linda Cole	IR Program Manager
Matt Audet	USEPA RPM	USEPA	Contact Linda Cole	Contact Linda Cole	RPM
Iver McLeod	MEDEP PM	MEDEP	Contact Linda Cole	Contact Linda Cole	RPM
Gail Lipfert (1)	MEDEP	MEDEP	Contact Linda Cole	Contact Linda Cole	Geologist
Debbie Cohen	Tetra Tech Facility Coordinator	Tt	Contact Linda Cole	Contact Linda Cole	Facility Coordinator
Lisa Joy (1)	PWD-ME Env, Director	Navy	Contact Linda Cole	Contact Linda Cole	PWD Env, Director
Tim Smith (1)	Tetra Tech Civil Engineer	Tt	Contact Linda Cole	Contact Linda Cole	Project Engineer
Dan Witt (2)	Tetra Tech Project Manager	Tt	Contact Linda Cole	Contact Linda Cole	Project Manager

(1) Attended the December 8, 2009, meeting only
(2) Participated in the March 23, 2010, conference call only

Comments/Decisions:

The project team reviewed the DQOs and information provided in draft versions of Worksheet #s 10 and 11 prepared to support DQO discussion. The following provides the major concerns and decisions related to those concerns. A more detailed meeting summary is provided in Appendix D.

- The CSM should reflect that contamination is likely a series of hotspots based on past operations.
- The CSM should reflect that underlying soil would not be expected to be impacted if the surface soil is not impacted.
- The total PCB calculation method considering the homolog analysis should be added to the SAP.
- The pre-design sampling boundaries were discussed, and it was agreed that the lateral limits of OU2 would not be extended beyond the proposed sampling limits.
- MEDEP expressed a concern with how confidence bands and variability in the data were being accounted for in the comparison of the data to the PALs.
- During the March 23 conference call, the project team discussed and agreed that the method to address confidence in determining areas requiring an action would be similar to the method used and accepted in the Feasibility Study.
- Two general methods of analyzing samples were discussed: vertical phasing and horizontal phasing of the soil analyses. In vertical phasing, the need to analyze soil samples from the deeper intervals at a particular soil boring is based on the sample results from the upper intervals (i.e., a step down approach). In horizontal phasing, the need to analyze soil samples further from areas of known contamination at a particular soil boring is based on the sample results from the soil borings closer to areas of known contamination (i.e., a step out approach). It was agreed that vertical phasing of soil analysis would be more appropriate than horizontal phasing based on the CSM. Based on vertical phasing, it was decided that all samples from 0 to 2 feet bgs would be analyzed first, followed by deeper soil analysis if the upper sample showed contamination.

Action Items:

- Show wetlands function and value evaluation area and show where the sediment intertidal area is located in a site figure.
- Add to CSM why antimony is not a primary COC.
- Include methodology for calculation of total PCBs using the homolog data.
- Revise text to reflect vertical phasing of analysis of samples.
- Propose a method to evaluate the confidence of the sample data when comparing sample results to DQO action levels.

Consensus Decisions:

- Vertical phasing of sample analysis is an appropriate methodology to focus data collection for this project.
- The pre-design study area boundary will represent the maximum potential horizontal extent of OU2.
- The method to address the confidence in determining areas requiring action was discussed during the March 23, 2010, conference call, and it was accepted by the project team.

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

10.1 SITE LOCATION AND DESCRIPTION

PNS is a military facility with restricted access located on an island in the Piscataqua River, as shown on Figure 1. The Piscataqua River is a tidal estuary that forms the southern boundary between Maine and New Hampshire. PNS is located in Kittery, Maine, north of Portsmouth, New Hampshire, at the mouth of the Great Bay Estuary (commonly referred to as Portsmouth Harbor). PNS is engaged in the conversion, overhaul, and repair of submarines for the Navy.

OU2 is located in the south-central portion of PNS as shown on Figure 1. OU2 consists of Site 6 – DRMO Storage Yard, Site 29 – Former Teepee Incinerator Site, and the DRMO Impact Area. Past investigations (described below) have shown that various chemical contaminants have been released at the DRMO as a result of site activities. The OU2 layout, including approximate site boundaries, is shown on Figure 2. Based on the evaluation of site uses and contaminant distribution, Site 6 and portions of Site 29 are referred to as the DRMO area, and the remainder of Site 29 is referred to as the Waste Disposal Area (WDA) in the OU2 FS Report (Tt, November 2008). This SAP follows this naming convention for consistency. Chemical contamination at OU2 was delineated for residential, occupational, and construction worker receptors for all of OU2 but not for the area west of OU2 where contaminants may also have been released as a result of OU 2 activiites. Soil chemical data from the area west of OU2 are needed to complete the OU2 RD; therefore, this SAP and the corresponding CSM focus on the area west of OU2 (Figure 3).

10.2 SITE HISTORY

The area western end of OU2 was originally known as Henderson's Point, which was a portion of land that was removed in the early 1900s. Quarters S and N (residential land use) were located near the historical (1901) shoreline before the adjacent area was filled to extend the size of the island. The majority of the filling in the area was conducted between 1902 and 1908 with material from the excavation of Henderson's Point. The excavated material from Henderson's Point included excavated soil, gravel, and blast rock and wood from a cofferdam. Other debris (including material such as wood from removed structures) generated during the excavation activities was also included in the fill material.

The DRMO, also located in the western end of OU2, was established in 1920. Materials reportedly stored at the DRMO included lead and nickel-cadmium battery elements, motors, typewriters, paper products, and scrap metal. Historically, DRMO operations primarily appear to have occurred in the current fenced area of the DRMO, but operations could have occurred in adjacent areas.

The area west of OU2 (see Figure 3), which includes John Paul Jones Avenue, has been used to access the DRMO area since the DRMO operations began. When railroad lines were used to transport materials to and from the DRMO, loading and offloading of materials for storage at the DRMO also occurred in the area south of Building 348, near the DRMO entrance.

Building 233 was located west of the DRMO area near the quay wall along Berth 7 circa 1957 to 1998; therefore, during that time period, DRMO operations and corresponding potential contamination would not have impacted the area of Building 233. Figure 3 shows a detail of the western portion of the DRMO area and the former locations of Building 233 and rail lines in this area.

10.3 HISTORY OF ENVIRONMENTAL INVESTIGATIONS

The Navy has investigated site hydrogeology, assessed the nature and extent of contamination, and performed risk assessments at OU2. OU2 is currently in the RI/FS stage of the CERCLA process. Environmental samples were collected at OU2 as part of various investigations including the Final Confirmation Study (FCS) in 1984 (LEA, June 1986), Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) from 1989 to 1992 (McLaren/Hart, July 1992), RFI Data Gap Investigation in 1994 (Halliburton NUS, November 1995), groundwater monitoring from 1996 to 1997 (Tt, August 1999), Field Investigation at Site 29 in 1998 (Tt, March 2000), removal action at Site 6 in 1999 (FWENC, June 2001), soil washing treatability study in 2004 and 2005 (Tt, January 2006), and additional investigation in 2007 and 2008 (Tt, August 2008). Soil samples were also collected to support a shipyard utility trench excavation for Building 298 in 2002 (Tt, November 2005). Samples were collected in the offshore area of OU2 [DRMO Storage Yard Area of Concern (AOC)/MS-11] as part of the Estuarine Ecological Risk Assessment (EERA) in 1991 to 1993 (NCCOSC, May 2000), Interim Offshore Monitoring Program from 1999 to 2003 (Tt, November 2004), and Additional Scrutiny Investigation in 2005 (Tt, August 2007).

Based on evaluation of the results of these investigations, a current understanding of contaminant sources, contaminant transport potential, representative receptors that could be exposed to contaminants under certain conditions, and the pathways by which these receptors could be exposed in the area west of OU 2 has been developed. A CSM describing the relationships among these factors is presented in Section 10.4.

10.4 CONCEPTUAL SITE MODEL

The CSM for the pre-design sampling focuses on potential sources, contaminants, potential receptors, and exposure pathways for soil in the area west of the OU2. Other site media (i.e., groundwater, surface water, sediment) are not considered to pose unacceptable risks to human or ecological receptors (Tt,

March 2010). The CSM for the area west of OU2 is summarized on Figures 4a, 4b, and 4c and in the text below.

10.4.1 Geology

Limited geologic and hydrogeologic information is available for the pre-design study area. The hydrogeology of this area is based on information from the OU2 Supplemental RI Report (Tetra Tech, March 2010) and boring data.

The islands composing PNS are formed on bedrock highs with thin veneers of glacial till, recent alluvium, and fill materials. Based on the historical shoreline, most of OU2 was created by filling; however, the pre-design study area lies with the limits of the original island. The majority of the shoreline of the pre-design study area was created when Henderson Point was blasted away in 1905 and is bedrock outcrop of a dark gray or greenish-gray quartzite. Subsurface materials in the area of study are anticipated to include the following (from lowest to highest elevation): bedrock, weathered bedrock, and fill. A generalized cross section of the subsurface is illustrated in Figure 4A.

A limited number of geotechnical borings related to the construction of Building 348 and environmental borings related to the OU2 Supplemental RI Report have been installed within the pre-design study boundary. The borings around Building 348 encountered refusal on bedrock or boulders at approximately 10 to 13 feet below ground surface. The overburden was reported to be sand and gravel fill with lesser amounts of loam, peat, silt, and clay. Environmental borings along the shoreline (OU2-174 and OU2-124) in the westernmost portion of OU2 encountered sandy fill overlying weathered bedrock to depths of 2 to 3 feet and bedrock at approximately 4 feet. A topographic map from 1901 (prior to the removal of Henderson Point) shows the vicinity of Building 348 being in a low area between Henderson Point and the Seavey Island. This area was likely filled (presumably with rock fill from Henderson Point) to current grade. Therefore, it is anticipated that the bedrock surface is deeper near Building 348 than at the shoreline. Back channel-type deposits (e.g., peat and slit or clay) may be encountered below the surface fill in this historically topographic low area.

No monitoring wells have been installed within the pre-design study boundary; therefore, the hydrogeology of this area is interpreted based on the known hydrogeology of PNS and OU2. Groundwater at PNS generally flows from the original island interiors toward the current coastline. The overall conceptual model of groundwater flow at the facility is that fresh groundwater originating from precipitation in the island interior flows downward and outward through both the overburden and bedrock. Tidal effects are present near the shorelines with a net groundwater flow outward into the Piscataqua River.

The depth to groundwater is estimated to be approximately 10 feet below ground surface in the pre-design study area at high tide with the tidal range of approximately 8 feet. Based on these depths, the groundwater is expected to occur in the bedrock along the shoreline and in the filled area near Building 348. Groundwater is expected to occur in the bedrock North of Building 348, based on topography.

10.4.2 Potential Sources of Contamination

The OU2 Supplemental RI Report (Tt, March 2010) identified for OU2 soil include antimony, copper, nickel, lead, PCBs, and polycylic aromatic hydrocarbons (PAHs), and dioxins/furans as the COCs for soil. As explained in the Final OU2 Supplemental RI Report, the maximum extent of the soil COCs are within the areas defined by copper, lead, and PCB contamination (Tt, March 2010). Because contaminant release mechanisms in the area west of OU2 and within OU2 were similar, and subsequent migration potential is also similar, the relative spatial distributions of contaminants in these two areas is assumed to be similar. Therefore, copper, lead and PCB concentrations in the area west of OU2 are necessary to delineate contamination because previous OU2 soil data indicate that unacceptable risks due to soil with contamination from the other COCs are encompassed within areas of unacceptable risk delineated by copper, lead, and PCB contamination.

For the Revised Draft FS Report, lead, copper, and PCB concentrations were used to delineate contamination for residential, occupational worker, and construction worker receptors. Therefore, lead, copper, and PCBs are the only COCs that will be used to delineate contamination in the area west of OU2.

The contaminant sources in the DRMO area resulted from historical use of portions of the area as a storage yard. Specifically, the following are identified as the OU2 sources, release mechanisms to soil, and COCs associated with OU2 sources as provided in the OU2 Supplemental RI (Tt, March 2010):

- Former battery storage in the DRMO area (before 1983): Open storage of lead-acid batteries (plates and other battery elements stockpiled on uncovered pallets) occurred primarily at two locations in the DRMO area (areas capped/asphalted during the 1993 interim action). Nickel-cadmium battery elements were also reportedly stored in the same manner and areas. Open storage of batteries was discontinued in 1983. Degradation (weathering) of the lead-containing materials from the batteries and possible leaching of the lead or leakage of acid electrolyte during storage are the primary release mechanisms from this previous potential contaminant source.

- Equipment and material storage at the DRMO area (before 1983): Industrial equipment and materials made of metals, plastics, fabric, wood, and glass and possibly containing tars, oils, solvents, and other chemicals were stored in unpaved portions of the DRMO area. Possible leaks or spills from the

equipment or materials to soil likely have occurred based on detections of PCBs at concentrations greater than approximately 10 mg/kg in soil in the south-central portion of the DRMO area (Tt, March 2010). PCB concentrations greater than 10 mg/kg detected in the DRMO area are not adjacent to the study area for this investigation and therefore are not shown on figures in this document.

- Other potential OU2 contaminant sources include loading and offloading of equipment and materials for storage in the DRMO area (before 1983), which involved the same chemicals as those listed for the previously described sources. Loading and offloading of equipment and materials may also have contributed to contaminated soil within the DRMO area and in the area south of Building 348, near the DRMO entrance. Loading and offloading operations would have most often occurred near the entrance to the DRMO area but occasionally could have occurred farther away depending on the operations at that particular time. Because of the intermittent and changing nature of the loading operations, it is anticipated that OU2 soil contamination west of the current OU2 boundary would be sparse. It is likely that greater levels of contamination would exist closer to the entrance of the DRMO area because items would have been more routinely offloaded there. Contamination deposited on the ground surface may have leached to the subsurface via infiltration of precipitation. Lead, copper, and PCBs are relatively immobile in soil; therefore, concentrations of these contaminants are expected to decrease with depth.

Copper slag may be located within the study boundaries because slag with copper concentrations greater than 10,000 mg/kg was found in the western portion of the DRMO area (within the current OU2 boundary). The slag was found to extend to approximately 2 to 3 feet bgs. Slag was also found in a portion of the DRMO where batteries were stored (Tt, March 2010).

Before soil at the DRMO was covered with either an interim engineered cap or pavement, physical movement of contaminated soil (e.g., through snow plowing) was a secondary release mechanism that could have resulted in contamination of areas adjacent to the DRMO, including the area to the west. The snow was historically plowed over the shoreline into the Piscataqua River or into piles near the entrance of the DRMO (PNS, January 1997). Currently, the DRMO is paved and snow is piled within the DRMO.

One surface soil sample (SS-24) was located to the west of DRMO near the quay wall in a narrow strip of land between the quay wall and the former location of Building 233 (See Figure 3), which was in existence from the 1950s through the 1990s. Building 233 is noted as a radiological decontamination training facility in the 1957 map of the shipyard and then the building is listed as production storage on 1979, 1982 and 1998 historical maps of the shipyard. Sample SS-24 was collected as part of the 1989 to 1992 RFI investigation when Building 233 was in existence. Although sample SS-24 contained lead at

2,830 mg/kg, it is unlikely that OU2 operations (offloading and/or snow plowing) would have occurred closer to the shoreline because of the presence of Building 233 and the quay wall. There would be no reason to offload materials between the quay wall and Building 233 for OU2 activities. Therefore, the area around SS-24 is not included as part of the study area west of OU2.

Historical use of railroads and current and historical use of asphalt-paved roads likely contributed to chemical concentrations (particularly lead) detected at and in the vicinity of OU2. However, fill material placed during filling activities from approximately 1902 to 1908, which is not considered a site contaminant source, may have been impacted by one or more of the sources identified above such as the historical use of railroads and current and historical used of asphalt-paved roads.

10.4.3 Potential Exposure Routes and Receptors

The area west of the DRMO area is used by occupational workers. Current exposure would be for an occupational worker exposed to surface soil (0 to 2 feet bgs) or a potential construction worker exposed to surface and subsurface soil (0 to approximately 10 feet bgs, which roughly cooresponds to the top of the tidally saturated zone in this area). The 0 to 10 foot bgs depth was used to span they zone for human health exposure in the OU2 Supplemental RI. A site construction worker may also be exposed to groundwater. Site groundwater data indicate that even if COCs are migrating to groundwater from soil, the concentrations of those COCs in groundwater are not great enough to produce unacceptable risk to human health or the environment. Furthermore, the sources of contamination in the study area originate from DRMO activities that took place over 25 years ago; therefore, sufficient time has passed to determine whether those COCs are migrating from soil to groundwater. Additional evaluation of the soil-to-groundwater migration pathway is not included as part of this investigation because groundwater data do not indicate unacceptable risks to human health or the environment, and COC contamination has been in place long enough to detect the effects of soil-to-groundwater migration.

Risks to occupational workers exposed to surface soil via direct contact (i.e., dermal contact, ingestion of soil, and inhalation of soil particles) would be of concern if pavement or vegetation was removed. Although recreational users could access these areas, the areas are not maintained for recreational use. Future residential use of the area could only occur under a potential future site development scenario. Details on the routes of exposure are shown on Figures 4B and 4C for human health and ecological receptors, respectively.

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

11.1 PROBLEM DEFINITION

Chemical analysis of soil to define the limit of soil contamination on the western side of OU2, a wetlands functional value and habitat assessment of the OU2 intertidal zone, and topographic mapping of OU2 are required to support a RD. The information gained from these activities will be provided to the RD engineers to help them design a cost-effective remedy to reduce human health risks at OU 2. Delineation of contamination will be estimated based on evaluation of chemical analysis data.

11.2 INFORMATION INPUTS

The following information is required to complete the RD:

11.2.1 Soil Analyses

Analytical Data - Requirements for soil sampling and analysis methods are described below. Additional details [e.g., the exact methods and Standard Operating Procedures (SOPs) to be used] are provided in Worksheet #s 15, 19, and 21. Required analyses are presented in Worksheet #18. The analytical methods (see Worksheet #19) were selected to be the same as those used previously or at least comparable to previously used analytical methods with respect to precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS). Limits of Quantitation (LOQs) were selected to be less than PALs.

- **Sampling Methods** - Sampling methods were selected to be the same as those used previously or at least consistent with the representativeness and comparability of soil samples collected previously. Direct push technology (DPT) is the most cost-effective method for acquiring soil samples. Worksheet #18 lists sampling methods.

- **Project Action Levels -** PALs (see Worksheet #15) for this investigation are the OU2 Preliminary Remediation Goals (PRGs) for copper, lead, and PCBs. These PRGs were developed in the Revised Draft OU2 FS Report (Tt, November 2008), separately for each potential receptor (i.e., hypothetical future resident, occupational worker, and construction worker). The PRGs are chemical specific goals for representative site concentrations (based on the exposure concentration) that when achieved will result in site-wide concentrations that pose an acceptable risk for the targeted receptor. As described in the FS Report (Tt, November 2008), all but the OU2 lead PRGs are based on a 5×10^{-6} carcinogenic risk or a Hazard index (HI) of 1 for non-carcinogens. The lead

PRG is based on lead exposure modeling described in Attachment 1 of Appendix A of the OU2 FS Report (Tt, November 2008).

The residential, occupational, and construction worker OU2 PRGs, presented on Worksheet #15, do not necessarily indicate the final concentrations remaining after remediation. If the PRGs are not achieved, however, land use controls (LUCs) may be required to protect potential receptors from exposure to residual contamination, depending on the selected remedy. The project occupational and construction action levels are levels that if exceeded require consideration of soil remediation under an occupational or construction land use scenario.

11.2.2 Topographic Survey

A topographic survey must be performed on the OU2 area to provide the required base mapping for the RD. The survey must be completed to a scale of 1 inch equals 50 feet and a contour interval of 1 foot. The survey must be completed by a Licensed Maine Professional Land Surveyor in accordance with the National Society of Professional Surveyors model standards for topographic surveys.

11.2.3 Wetlands Functional Value and Habitat Assessment.

The wetlands functional value and habitat assessment must be completed following the methods described in the United States Army Corps of Engineers document The Highway Methodology Workbook Supplement, Wetlands Functions and Values: A Descriptive Approach (United States Army Corp of Engineers, September 1999).

11.3 DELINEATION OF STUDY BOUNDARY

Two soil populations must be represented by data collected during this investigation. The population of primary interest is surface and subsurface soil contaminated by DRMO operations. The other population is surface and subsurface soil not contaminated by DRMO operations. Contamination is defined as soil COC concentrations in excess of the OU2 PRGs for residential, occupational, or construction worker receptors, as applicable (See Section 11.4).

The geographical/physical boundary of OU2 is illustrated on Figure 2. The study area boundary, which is the area to the west of the DRMO area, is shown on Figure 5.

Although contamination may have been spread to the offshore area during past snow-plowing activities the offshore area is being investigated as part of OU4 and therefore is not included in this investigation. The shore line in the study boundary consists of either bedrock outcrops, seawalls or revetment and does

not consist of any soil on the surface. The pre-design study boundary and the rationale for choosing boundary limits are as follows:

- The eastern lstudy area boundary is the current OU2 boundary presented in the FS. The OU2 boundary (shown on Figure 5) runs along the DRMO fence line and also includes an area extending west from the DRMO between John Paul Jones Avenue and the shoreline.

- The study area for this project does not include the area just west of the OU2 boundary where sample results have shown the soil does not contain concentrations of COCs greater PRGs (Sample locations OU2-159 and OU2-158).

- The southwestern study area boundary extends from the current OU2 boundary along the shoreline to the southern end of the quay wall. The end of the quay wall also roughly corresponds to the end of the former location of Building 233. Building 233 was in existence from the 1950s through the 1990s. The boundary is located here because it is unlikely that OU2 operations (offloading and/or snow plowing) would have occurred further along the shoreline because of the presence of Building 233 and the quay wall. There would be no reason to offload materials between the quay wall and Building 233 during OU2 activities.

- The western study area boundary extends from quay wall across John Paul Jones Avenue and follows the edge of the road to the north. The study boundary is approximately 100 feet from the current OU2 boundary along the road. It is thought that offloading operations would not have routinely occurred farther from the entrance to the DRMO.

- The northern study area boundary approximately follows the base of a hill in this area (as bounded by the 110-foot contour (2002 PNS Vertical Datum) because it is unlikely that materials would have been offloaded on the side of the hill or that snow would have been plowed up the hillside. The study area boundary follows the 110-foot contour until this contour starts turning north to parallel John Paul Jones Avenue. The boundary then turns and goes to the edge of Jone Paul Jones Avenue.

- The vertical study boundary includes soil form the ground surface to an approximate depth of 10 feet bgs or bedrock, whichever is shallower. The 10-foot depth approximates the top of the tidally saturated zone and was also used to define the zone of potential human exposure in the Draft Final OU2 Supplement RI Report (Tt, March 2010).

The study area is approximately 11,000 square feet or 0.25 acre. Because this area has been an industrial area for over 100 years it is possible that contamination exists outside the study boundary;

however, due to the presence of physical boundaries that have existed for decades to limit the spread of contamination from OU2 and based on knowledge about the locations of past OU2 operations, it is unlikely that this contamination would be associated with the DRMO area. If contamination is found at the edge of the study boundary, additional investigation of this contamination will not be completed as part of OU2 investigations. The data collection density must be sufficient to assure the project team that any significant contamination from OU2 in the western area will be delineated.

11.4 DEVELOP DECISION RULE

Because the wetlands assessment and topographic survey will be provided for informational purposes; to the design engineers, there are no decisions to be made for this investigation based on those data. Decision rules were, however, developed to govern data use for soil and are presented below. For each rule, multiple, noncontinguous areas of contamination within the study boundary may be delineated to account for all contamination exceeding action levels. Project Action Levels (PALs) to which the data are compared are presented on Worksheet #15.

Decision Rule #1 (Lateral extent of surface contamination):

If all surface soil concentrations for the chemicals listed in Worksheet #15 obtained during this pre-design investigation are less than or equal to the residential PALs, do not change the limit of contamination presented in the FS. If any surface soil concentration is greater than the residential PAL within the study boundary the project team will review the spatial distribution of the contamination, the magnitude of the contamination, the concentration gradients between samples, and past operations within the study boundary. Based on this review, the project team will agree on the area(s) within which concentrations exceed the residential project action levels and area(s) within which the lower of construction worker or occupational worker PALs are exceeded.

The limit of contamination will not extend beyond the lateral study boundary because the study boundary was based on physical boundaries and on past operations within the study boundary such that any contamination outside the study boundary is unlikely to be attributed to OU2 as discussed in Section 11.3.

The project team assumes that for any area that has surface soil contamination in excess of the residential PALs but less than the lower of construction worker or occupational PALs, the likely remedy will be land use controls. These controls will extend vertically from the surface to include all the unsaturated soil, therefore, delineation of subsurface soil above the residential PAL but less than the lower of the construction worker or occupational worker PALs is not required,

Decision Rule #2 (Vertical extent of contamination):

Note: this rule applies only in those locations where surface contamination exceeds the lower of construction worker or occupational worker PALs. These locations are referred to as selected locations.

If all subsurface soil contaminant concentrations in the selected location(s) is less than or equal to the lower of the occupational and construction worker PALs for all target analytes, conclude that contamination is limited to the surface at the selected location. If any suburface soil concentration is greater than either of the occupational or construction worker project action levels, the project team will review the spatial distribution of the contamination, the magnitude of the contamination, the concentration gradients between samples, depth of contamination, and past operations within the study boundary. Based on this review, the project team will develop the vertical extent and depth of contamination that requires a remedial action under the occupational or construction worker risk scenario.

11.5 PERFORMANCE CRITERIA

The project team selected locations and numbers of samples that, based on their experience, support the attainment of project objectives. The project team must use the results of this investigation to determine whether the amount, type, and quality of data collected are sufficient to support the RD. When reviewing the target analyte delineation data, the project team (including the Navy, USEPA, and MEDEP) must consider the spatial distribution of contamination, contaminant concentration gradients between samples, and understanding of how contaminants were deposited or moved in the study area (conceptual model). The project team must then discuss and agree on any areas that require an action (such as placing LUCs or excavation/treatment of soil). The project team must account for uncertainty in this process by expanding areas where greater uncertainty exists (but not beyond the study boundary, because contamination beyond the study boundary is not attributable to DRMO operations, as discussed above in section 10.4.1). For instance, if an area had a mix of true high and low sample results (some greater than and some less than an action level), the team could decide to account for the uncertainty in the area by including the entire area of measurements within the limit of contamination so that the team is confident the contamination is well bounded.

Results of data validation (i.e., data qualifications) must be considered in these evaluations, with an increased tendency to recommend additional investigation with decreased data quality. The data usability evaluation process is described in more detail in Worksheet #37.

11.6 PLAN FOR OBTAINING DATA

The plan for obtaining the data for the OU2 Pre-Design Investigation is described in detail in Worksheet #17 along with the sampling designs and rationales.

SAP Worksheet #12 -- Measurement Performance Criteria Table

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table – Field QC Samples –Soil

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Blanks	All Fractions	One per source ofwater.	Accuracy/Bias/ Contamination	No analytes ≥ ½ Quantitation Limit (QL), except common laboratory contaminants, which must be < QLs.	S&A
Equipment Rinsate Blanks	All Fractions	One per 20 field samples per matrix per sampling equipment[1].	Accuracy/Bias/ Contamination	No analytes ≥ ½ QL, except common laboratory contaminants, which must be < QLs.	S&A
Field Duplicates	All Fractions	One per 10 field samples collected.	Precision	Values > 5 times QL: Relative Percent Difference (RPD) ≤ 50% (soil)[(2),(3)]	S
Cooler Temperature Indicators	All Fractions	One per cooler.	Representativeness	Temperature between 2 and 6 degrees Celsius (4 ± 2 °C).	S

1 – Equipment rinsate blanks will be collected if non-dedicated submersible pumps or other equipment are used.
2 – If duplicate values for non-metals are less than five times the QLs, the absolute difference should be greater than two times the QLs.
3 – If duplicate values for metals are less than five times the QLs, the absolute difference should be less than four times the QLs.

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table

(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title, and date)	Data Generator(s) (originating organization, data types, data generation/collection dates)	How Data Will Be Used	Limitations on Data Use
Revised Draft Feasibility Study for Operable Unit 2	Tt, November 2008	Tt, soil data, 1989 to 2007.	Soil data adjacent to the study area of this investigation may be plotted on a map with data generated during this investigation to delineate soil contaminant extent.	None.

SAP Worksheet #14 -- Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

Summary of Project Tasks

Sampling Tasks:

- Mobilization/demobilization
- Site-specific health and safety training
- Utility clearance
- Obtain sampling coordinates. A global positioning system (GPS) unit or the measured distance (e.g., using a tape measure) from known site features will be used to record sampling locations and obtain sampling coordinates.
- Monitoring equipment calibration
- DPT (19 borings) soil sampling (see Figure 5)
- Equipment decontamination
- Investigation-derived waste (IDW) will be drummed by Tt. PNS will provide the appropriate IDW containers, and PNS is responsible for sampling, analyzing, and disposing of IDW in accordance with state and federal guidelines.
- Field QA/QC management
- Field documentation QA/QC and review
- Sample custody and shipment

Analysis Tasks:

- The laboratory will analyze soil samples for PCBs (homologs), copper, and lead as outlined in Worksheet #15. Total PCB homologs will be calculated by the laboratory per sample by summing all 10 of the PCB homolog groups.

Quality Control Tasks:

- Implement SOPs for sampling activities, sample preservation/shipping methods, and sample preparation/analysis methods in accordance with the SAP.
- Collect and analyze QA/QC field samples including duplicates, equipment rinsate blanks, and temperature blanks (for coolers).
- Field duplicate samples will be collected following the same sampling procedure as environmental samples.
- Laboratory analysis of matrix spike/matrix spike duplicate (MS/MSD) and laboratory duplicate samples.
- Calibration and other QC checks as specified in Worksheet #s 24 and 28.

Data Management Tasks:

- Data Receipt and Electronic Data Deliverable (EDD) upload (Sample Management Coordinator)
- Data verification and validation (Data Validation Chemist or Environmental Scientist and DVM)
- Upload of validated data to the project database (Project Database Manager) and Naval Installation Restoration Information Solution (NIRIS).

Documentation and Records:

- Documentation of sample location coordinates, borings logs, chain-of-custody forms, sample logs, and shipping documents for all samples.
- The EDDs and corresponding databases will be formatted in both Navy and MEDEP formats.
- Preparation of electronic and hardcopies of the finalized OU2 Pre-Design Investigation.
- Upload of the finalized OU2 Pre-Design Investigation SAP to NIRIS

Data Packages:

- Generation of Tt data validation reports

Data Review Tasks:

- Data verification and validation will be performed by Tt as provided in Worksheet #s 35 and 36. A data validation report will be produced for each Sample Delivery Group (SDG).

- All field data records and validated data will be reviewed by Tt to determine the usability of the data. The outcome of this assessment will be conveyed to the project team for agreement before the project report is finalized. Data limitations pertaining to project quality objectives and action limits will be identified, and corrective actions will be taken as necessary. Data usability will be assessed in accordance with the process describe in Worksheet #37.

SAP Worksheet #15 -- Reference Limits for Soil

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil

Analytical Groups: PCBs, Copper, Lead

Analyte	Chemical Abstract Service (CAS) Number	Screening Level (mg/kg)	Reference	Screening Level (mg/kg)	Reference	Screening Level (mg/kg)	Reference	Project LOQ Goal (mg/kg)	Mitkem Detection Limits(1)	
									LOQ (mg/kg)	LOD (mg/kg)
Metals										
Copper	7440-50-8	51,600	OU2 Construction Worker PRG	68,100	OU2 Occupational PRG	7,300	OU2 Residential PRG	2,433	1.5	0.75
Lead	7439-92-1	2,000	OU2 Construction Worker PRG	1,600	OU2 Occupational PRG	400	OU2 Residential PRG	133	0.5	0.25
PCB (homologs)										
TOTAL PCBS	NA	155	OU2 Construction Worker PRG	6.0	OU2 Occupational PRG	1	OU2 Residential PRG	0.33	0.00042	0.00033

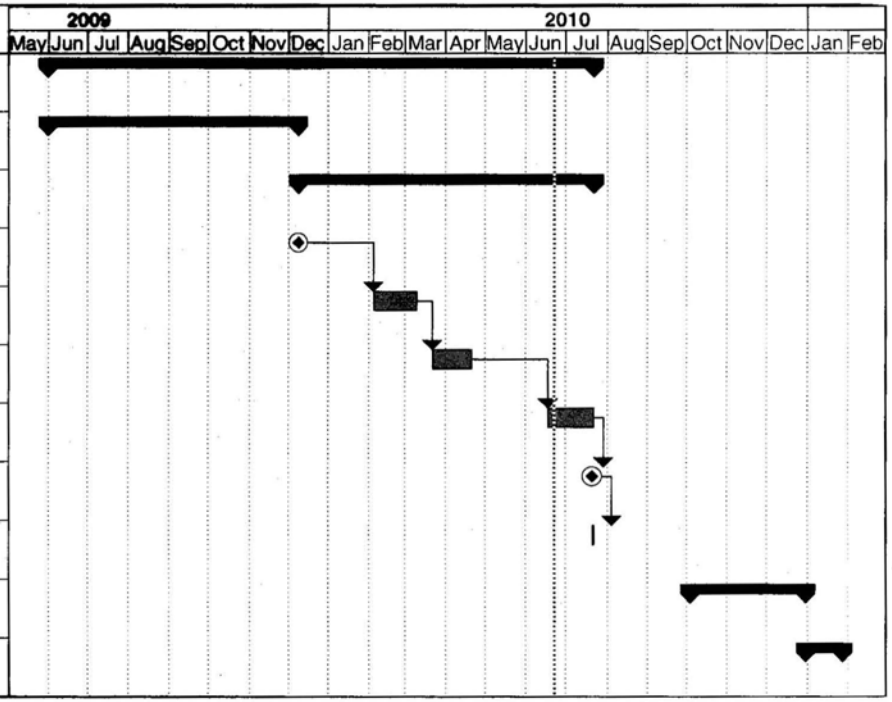
LOD = Limit of Detection
LOQ = Limit of Quantitation

1 Mitkem detection limits are current as of May 2010.

SAP Worksheet #16 -- Project Schedule/Timeline Table

(UFP-QAPP Manual Section 2.8.2)

Task	CTO Plan Begin	CTO Plan End
PRE-DESIGN INVESTIGATION	Mon 6/1/09	Wed 7/21/10
DQOs	Mon 6/1/09	Wed 7/15/09
OU-2 Pre-Design Work Plan	Fri 12/4/09	Fri 10/8/10
Submit Draft Work Plan	***Tue 7/6/10***	***Tue 7/6/10***
Navy, USEPA & MEDEP Review	Wed 7/7/10	Fri 8/6/10
Navy/Regulators Comment Resolution	Mon 8/9/10	Tue 9/7/10
Prepare Final Work Plan	Tue 9/7/10	Fri 10/8/10
Submit Final Work Plan	***Fri 10/8/10***	***Fri 10/8/10***
USEPA, MEDEP & RAB Receive Final Work Plan Report	Fri 10/8/10	Fri 10/8/10
FIELDWORK	Tue 10/12/10	Fri 1/7/11
Submit Data Package	Mon 1/10/11	Fri 2/4/11



Project Manager:
Date: Tue 6/22/10

Task		External Tasks		External Milestone	
Progress		Project Summary		Deadline	
Milestone		Split			
Summary		Rolled Up Split			

SAP Worksheet #17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

The OU2 sampling strategy consists of a judgmental sampling design to define the limit of soil contamination on the western side of OU2, in the vicinity of Building 348 and John Paul Jones Avenue. Soil borings will be spaced approximately 25 feet apart across the study boundary to provide data to delineate remediation areas.

A boring must be placed between SS-01-03 and OU2-124, within the OU2 boundary shown in the FS Report (Tt, November 2008), to better delineate occupational and construction worker remediation areas within the current OU2 boundary. Another boring must be located near SS-02 to confirm high lead concentrations in this area.

Soil samples will be collected from all locations and depths during a single field event. The analysis of the samples will be completed in phases. All of the surface soil samples (0 to 2 foot sample) must be analyzed in the first phase. The decision to analyze a deeper sample will be based on the result of the sample result from the soil sample above it and will essentially be a step down approach. The results from the surface soil samples will be used to determine:

- the horizontal extent of contamination above residential PALs according to decision Rule #1 presented in Worksheet #11, and

- to determine if the next lower sample at a particular boring location should be analyzed based on the lower of the occupational or construction PAL.

That is, if the results of the first phase of soil samples sent for analysis are above the lower of construction worker or occupational PALs, then the next phase of samples at a boring location must be analyzed. The results from the surface and subsurface soil sample results will be used to determine the horizontal and vertical extent of contamination that will require a remedial action under the occupational or construction worker risk scenario according to decision Rule #2 presented in Worksheet #11.

Site Utility Clearance and Digging Permit

Before any intrusive drilling or subsurface sampling activities begin at the site, utility maps of the facility will be obtained and reviewed by the FOL. PNS will be responsible for conducting the utility survey and marking utility locations as necessary in the sampling area. PNS will also be responsible for obtaining access to sampling locations, including moving of any equipment or stored materials at the DRMO and adjacent areas. Tt will be responsible for pre-marking proposed sampling locations or indicating areas for

sampling. No intrusive activities (e.g., drilling or test pitting) will occur until the proposed areas intended for subsurface investigations have been cleared by PNS. The FOL will review Tt SOP HS-1.0 to ensure that the utility clearance meets the intent of this SOP.

Pre-defined coordinates based on the sampling locations shown on Figure 5 will be loaded into a sub-meter resolution GPS unit, and the GPS will be used to navigate to each of the planned soil boring locations. The locations will be field-marked and labeled. Based on prior field investigations at OU2, it is anticipated that some pre-marked proposed boring locations will need to be moved, primarily to provide proper utility clearance but also to mitigate health and safety concerns, site access issues (proper and safe maneuvering of the drill rig, orientation of the drill rig with respect to current site activities, etc.), and to avoid unmovable surface obstructions. Tt will pre-mark specific boring locations to support the utility clearance as needed. Tt personnel will be present as necessary when the utility surveyor maps the subsurface utilities to assess the need to move any of pre-marked proposed locations. If any location needs to be moved by a distance of 10 feet or more, Tt personnel will contact the Tt PM and Navy RPM prior to field activities to determine whether the point should be eliminated or whether an alternate location can be found.

Soil Investigation Equipment and Procedures

Data must be obtained using sampling techniques that have been used at OU2 in the past. OU2 Pre-Design Investigation soil borings will be completed by DPT. A Geoprobe® or other similar DPT device will be used because of the greater mobility and flexibility it can offer compared to conventional drilling methods. The DPT will also generate less IDW, minimize decontamination time, and minimize access space required relative to conventional drilling methods.

Surface and subsurface soil boring samples from the unsaturated/upper tidally saturated zone will be collected to evaluate the horizontal and vertical extent of contamination on the western side of OU2. Samples will be collected in 2-foot increments (0 to 2 feet, 2 to 4 feet, 4 to 6 feet, 6 to 8 feet, and 8 to10 feet) until a depth of 10 feet bgs is reached or shallower refusal is encountered. Nineteen soil borings will be spaced approximately 25 feet apart across the study boundary to provide data to delineate remediation areas (see Figure 5). The soil sample analyses will be conducted in phases based on analytical results of the surface and sub-surface soil samples. Samples from the 0-to-2 foot interval will be analyzed in the first phase, if required at a particular soil boring location; the next phase would include the 2 to 4 foot interval and the 4 to 6 foot interval. The last phase, if required, would include the 6 to 8 foot interval and the 8 to 10 foot interval.

Site Restoration

All completed borings will be backfilled with bentonite chips and patched with cement grout, cold-patch asphalt, or soil at grade to match as closely as possible the surrounding surface. Each boring location will be revisited after backfilling is completed to reapply soil, cement grout, or cold-patch, if necessary. Borings that have been relocated (following utility survey or other reasons during the investigation such as shallow refusal) will be identified by a wooden stake or nail driven into the center of each location. The locations and distances between the borings will be clearly explained in the sample collection logs. Each stake will have brightly colored flagging attached to increase visibility and will be labeled using a waterproof marker with the soil boring number as determined by the FOL. Nails will have brightly colored flagging attached to increase visibility, and asphalt will be labeled using marking paint with the soil boring number. Stakes, flags, and nails that are not flush with the surface will be retained only until post-investigation surveying is completed and then will be promptly removed.

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

(UFP-QAPP Manual Section 3.1.1)

Sampling Location/ID Number	Matrix	Depth	Analytical Group	Number of Samples[1] (identify field duplicates)	Sampling SOP Reference[2]
OU2-400 to 418/OU2-SS-400-0002 to OU2-SS-418-0002	Surface Soil	0 to 2 feet bgs	PCB Homologs Copper Lead	19 plus 2 duplicates	CT-04, HS-1.0 GH-1.5 SA-1.3 SA-2.5 SA-6.1 SA-6.3 SA-7.1
OU2-400 to 418/OU2-SS-400-0204 to OU2-SS-418-0204	Subsurface Soil	2 to 4 feet bgs	PCB Homologs Copper Lead	76 plus 8 duplicates	
OU2-400 to 418/OU2-SS-400-0406 to OU2-SS-418-0406	Subsurface Soil	4 to 6 feet bgs			
OU2-400 to 418/OU2-SS-400-0608 to OU2-SS-418-0608	Subsurface Soil	6 to 8 feet bgs			
OU2-400 to 418/OU2-SS-400-0810 to OU2-SS-418-0810	Subsurface Soil	8 to 10 feet bgs			

1 All surface soil samples will be analyzed. Analysis of subsurface soil samples at each location will be determined based on the surface soil results in accordance with the decision rules. Therefore all subsurface soil samples collected may not be analyzed. In addition DPT refusal may be shallower than 10 feet at some locations; therefore, the target depth is 10 feet or refusal, whichever is shallower.

2 SOP that describes the sample collection procedures.

SAP Worksheet #19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference	Containers (number, size, and type)	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[1] (preparation/ analysis)
Aqueous Field Quality Control	PCB Homologs	SW-846 3510C, EPA 680/ Mitkem 60.0054, 50.0051	2 x 1000-milliliter (mL) glass bottles	2 x 1000 mL	Cool to 4 (±2) °C	7 days until extraction/40 days to analysis
	Metals – Copper and Lead	SW-846 3005A/3010A /6010C, Mikem 100.0111/100.0003	250mL plastic jar	150 mL	Nitric acid to a pH<2; Cool to 4 (±2) °C	180 days to analysis
Soil	PCB Homologs	SW-846 3550C, EPA 680/ Mitkem 60.0054, 50.0052	4 - ounce glass jar	30 grams	4º C (-10ºC after received by the laboratory)	365 days to extraction /40 days
	Metals – Copper and Lead	SW-846 3050B/6010C, Mikem 100.0111/100.0104	4-ounce glass jar	10 grams	Cool to 4 (±2) °C	180 days to analysis

1 Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted

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

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations(1)	No. of Field Duplicates	No. of MS/MSDs (2)	No. of Source Blanks	No. of Equip. Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
Surface Soil	PCBs	19	2	1	0	0	0	22
	Copper and Lead	19	2	1	0	0	0	22
Subsurface Soil	PCBs	76	8	4	0	0	0	88
	Copper and Lead	76	8	4	0	0	0	88

1 All surface soil samples will be analyzed. Analysis of subsurface soil samples at each location will be determined based on the surface soil results in accordance with the decision rules. Therefore all subsurface soil samples collected may not be analyzed. In addition DPT refusal may be shallower than 10 feet at some locations; therefore, the target depth is 10 feet or refusal, whichever is shallower.

2 Although MS/MSD samples are not typically considered field QC samples, they are included here because location determination will be established in the field by the FOL. The final column does not count MS/MSDs because MS/MSDs are additional volumes of samples planned to be collected and not separate samples.

SAP Worksheet #21 -- Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
CT-04	Sample Nomenclature	Tt	NA	N	Refer to Appendix C for field SOPs
GH-1.5	Borehole and Sample Logging	Tt	Geological logging	Y	
HS-1.0	Utility Locating and Excavation Clearance	Tt	Remote subsurface sensing, magnetometer, ground-penetrating radar	N	
SA-1.3	Soil Sampling	Tt	DPT	Y	
SA-2.5	Direct Push Technology (Geoprobe®/Hydropunch™)	Tt	Macrocore sampler, probe sampling adaptors, Roto-hammer with bit, disposable acetate liners, cast aluminum or steel drive points, Geoprobe® sampler	Y	
SA-6.1	Non-Radiological Sample Handling	Tt	Sample bottleware, packaging materials, shipping materials	Y	
SA-6.3	Field Documentation	Tt	Field logbook, field sample forms, boring logs, soil sample log sheets	N	
SA-7.1	Decontamination of Field Equipment	Tt	Decontamination equipment, scrub brushes, phosphate-free detergent, deionized water	N	

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

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
Photoionization Detector (PID)	Calibration gas	Daily	Manufacturer's guidance	Troubleshoot problem or replace equipment in accordance with manufacturer's instructions	FOL	Operation according to manufacturer's instructions	The PID will be used to meet health and safety requirements, and any decisions based on PID readings will be made in accordance with the HASP.

SAP Worksheet #23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (1) (Y/N)
Mitkem 20.0003	Logging Workorders and Samples into Omega ME, 11/5/09, Rev. 4	NA	NA	NA	Mitkem Laboratories	N
Mitkem 30.0003	Sample Receipt, Storage, Tracking and Disposal, 10/21/08, Rev. 12	NA	NA	NA	Mitkem Laboratories	N
Mitkem 30.0024	Sample and Waste Disposal, 10/21/08, Rev. 8	NA	NA	NA	Mitkem Laboratories	N
Mitkem 50.0051	Organic Preparation of Aqueous Samples by Separatory Funnel (Method 3510C), 2/4/08, Rev. 1	Definitive	Aqueous PCB Homologs Extraction	NA	Mitkem Laboratories	N
Mitkem 50.0052	Preparation of Soil Samples by Sonication (Method 3550), 1/08, Rev.2	Definitive	Soil PCB Homologs Extraction	NA	Mitkem Laboratories	N
Mitkem 60.0054	PCB Homologs by GC/MS/SIM using EPA Method 680, 1/24/08, Rev. 0	Definitive	Aqueous and Soil PCB Homologs	Gas Chromatography/ Mass Spectrometery/ Selective Ion Monitoring (GC/MS/SIM)	Mitkem Laboratories	N
Mitkem 100.0111	Metals by ICP-AES, Method 6010, Rev. 10, 11/08	Definitive	Aqueous and Soil Metals	Inductively Coupled Plasma – Atomic Emmisions Spectrometery (ICP-AES)	Mitkem Laboratories	N
Mitkem 100.0003	Sample Preparation of Aqueous Samples by Acid Digestion for ICP, Method 3005/3010, Rev. 6, 5/06	Definitive	Aqueous Metals Digestion	NA	Mitkem Laboratories	N

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (1) (Y/N)
Mitkem 100.0104	Sample Preparation of Soils by Acid Digestion for ICP, Method 3050,Rev. 6, 4/06	Definitive	Soil Metals Digestions	NA	Mitkem Laboratories	N

SAP Worksheet #24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS PCB Homologs	Initial Calibration - A five-point calibration performed for all analytes.	Instrument receipt, instrument change (new column, source cleaning, etc.), when continuing calibration verification (CCV) is out of criteria.	Percent relative standard deviations (%RSDs) must be <20% with a maximum of 10% of the target analytes and/or surrogate compounds allowed %RSDs <50%. Relative Retention Times (RRTs) must meet ±0.06 RRT units for each compound and surrogate.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	Mitkem 60.0054
	Initial Calibration Verification (ICV)	Once after each initial calibration.	Percent Recoveries (%Rs) of individual compounds must be within 80-120%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a decafluorotriphenyl phosphine (DFTPP) tune.	Percent differences (%Ds) must be < 20% with a maximum of 20% of the target analytes and/or surrogate compounds allowed %Ds <50%.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS PCB Homologs	Tune verification DFTPP	Every 12 hours.	Homolog – Ions – Ratio Criteria MonoCB, 188-190, 2.50-3.50 DiCB, 222-224, 1.30-1.70 TriCB, 256-258, 0.80-1.20 TetraCB, 292-290, 1.10-1.50 PentaCB, 326-324, 1.40-1.80 HexaCB, 360-362, 1.00-1.40 HeptaCB, 394-396, 0.90-1.20 OctaCB, 430-428, 0.90-1.30 DecaCB, 498-496, 1.20-1.60	Check instrument performance, perform maintenance, retune the instrument and reanalyze tuning solution	Analyst, Department Manager	
ICP-AES Metals: Copper and Lead	Initial Calibration – one-point calibration.	At the beginning of each day or if QC is out of criteria.	One-point calibration per manufacturer's guidelines.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	Mitkem 100.0111
	ICV (Second Source)	Before beginning a sample run.	%Rs of true values must be within 90-110.	Do not use results for failing elements unless the ICV > 110% and the sample is < the LOQ. Investigate and correct problem.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
ICP-AES Metals: Copper and Lead	Calibration Blank	Before beginning a sample sequence.	No analytes detected > Limit of Detection (LOD).	Correct the problem, then reprepare and reanalyze.	Analyst, Department Manager	
	CCV	At the beginning and end of each run sequence and every 10 samples.	Results must be within 90-110 %R of true values	Check problem, recalibrate, and reanalyze any samples not bracketed by passing CCVs.	Analyst, Department Manager	
	Low-Level Calibration Check Standard (if using one-point initial calibration)	At beginning and end of run.	Results must be within 80-120%R of the true value.	Do not use results for failing elements unless LOQ recovery is > upper limit and sample result < LOQ.	Analyst, Department Manager	
	Interference Check Standards (ICSs - ICSA and ICSB)	At the beginning of an analytical run.	ICSA recoveries must be less than the absolute value of the LOD, and ICSB recoveries must be within 80-120 %R of the true value.	Correct the problem, then reprepare and reanalyze all affected samples.	Analyst, Department Manager	

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

(UFP-QAPP Manual Section 3.2.3)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[1]	SOP Reference[2]
GC/MS	Check for leaks, check gas supply, recondition or replace trap, clean injection port/liner and septa as needed.	PCB Homologs - Tune (Homolog Tune), CCV.	Monitor instrument performance via tuning mass criteria and CCV.	Tune daily, CCV after every 12 hours of operation.	Ion abundance within acceptance limits for tune, CCV %D ≤ 20.	As needed, replace connections, gas line filters, trap, or GC column. Clip column, replace injection port liner, clean injection port, clean source. Repeat tune, calibration, or CCV and any affected samples. See Attachment in SOP for more details.	Analyst, Department Manager	Mitkem 60.0054
ICP-AES	Perform leak test, change pump tubing, change torch and window, clean filters	Metals - Monitor instrument performance via CCB and calibration blank.	Monitor instrument performance via CCV and calibration blank .	Daily, after every 10 samples	All analytes within +/- 10%, no analytes > LOQ.	Replace pump tubing, replace torch and window, clean all filters.	Analyst, Department Manager	Mitkem 100.0111

SAP Worksheet #26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL or designee/Tetra Tech
Sample Packaging (Personnel/Organization): FOL or designee/ Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL or designee/ Tetra Tech
Type of Shipment/Carrier: Fed Ex
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians/Mitkem
Sample Custody and Storage (Personnel/Organization): Sample Custodians/Mitkem
Sample Preparation (Personnel/Organization): Extraction Lab, Metals Preparation Lab/Mitkem
Sample Determinative Analysis (Personnel/Organization): GC/MS Lab, Metals Lab/Mitkem
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 6 months from submittal of final data report
Biological Sample Storage (No. of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodians/Mitkem

SAP Worksheet #27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Tt CT-04 Sample Nomenclature, Tt SA-1.2 Surface Water and Sediment Sampling, Tt SA-1.3 Soil Sampling, Tt SA-6.1 Non-Radiological Sample Handling
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): Mitkem SOP 30.0003, Sample Receipt, Storage, Tracking and Disposal.
Sample Identification Procedures: Mitkem SOP 20.0003, Logging Workorders and Samples into Omega ME, 11/5/09, Rev. 4
Chain-of-Custody Procedures: Mitkem SOP 110.0026, Handling of Evidentiary Materials; SOP 110.0027, Documentation Policy and Procedures

SAP Worksheet #28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	**Aqueous QC/ Soil**
Analytical Group	**PCB Homologs**
Analytical Method/ SOP Reference	**EPA 680/Mitkem 60.0054**

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method Blank	One for each batch of up to 20 samples of similar matrix.	Contaminants in the method blank must be < ½ LOQ.	Investigate source of contamination. Rerun method blank. Re-extract associated batch of samples unless not detected for the contaminant.	Analyst, Supervisor Laboratory QAM, and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	Two per sample: 3,3',4,4'-TetraCB-C13 2,2',3,3',4,4',5,5'-OctaCB-C13	%Rs must meet be between 60 and 140%.	If sample volume available, re-extract. Report both if second successful analysis is outside of holding time or both fail QC criteria. Flag with * on Form 2.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)/LCS duplicate	One for each batch of up to 20 samples, if there is no MSD.	%Rs must meet be between 60 and 140. RPD ≤ 40.	Reanalyze once. If the LCS recoveries are high but the sample results are <QL, narrate. Flag with * on Form 3.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One for each batch of up to 20 samples.	%Rs must meet be between 60 and 140. RPD ≤ 40.	If recoveries are outside limits and surrogate and LCS criteria are met, note in narrative. If both the LCS and MS/MSD are unacceptable, check standard prep. Speak with PM regarding further action. Flag outliers with * on Form 3.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
Internal Standard (IS)	One per sample – 2,2',5,5'-TetraCB-C13	IS area -50% to +100% compared to IS from CCV; IS retention time window ±0.5 minute compared to CCV retention time.	Reanalyze affected samples. If similar results, report both runs. Flag outliers with an * on Form 8.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Aqueous QC/Soil**
Analytical Group	**Metals**
Analytical Method/ SOP Reference	**SW-846 6010C/7470A/ Mitkem SOP 110.0111**

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method Blank	One for each digestion batch of up to 20 samples.	Contaminants in the method blank must be < ½ LOQ.	Redigest and reanalyze, if sample concentration is >10X blank concentration, then narrate.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per digestion batch of 20 or fewer samples.	%R must be within 80 to 120% of the true value.	Redigest and reanalyze, if recovery is high and sample is <QL, then narrate.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per digestion batch of 20 or fewer samples.	Recovery should be within 80-120% of the true value if sample < 4x spike added. RPD should be within ≤ 20.	Perform post-digestion spike analysis, qualify data.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution (SD)	One SD (5x) is performed for each batch of 20 samples of the same matrix.	%D must be within 90 and 110.	Check instrument performance, qualify data.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Post-Digestion Spike	For any element that fails in the MS where the native sample concentration was <4x the spike amount.	%R must be within 75 and 125.	Check instrument performance, qualify data.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet #29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document / Record	Where Maintained
Sample Collection Documents and Records Field logbook (and sampling notes) Field forms (e.g., boring logs, sample log sheets, drilling logs, etc.) Chain-of-custody records Equipment calibration logs Photographs Field Task Modification Request forms Field Sampling SOPs	Tt Project File. Field forms, chain-of-custody records, Field Task Modification Request forms, and photographs will also be included in the field investigation data package.
Laboratory Documents and Records Sample receipt/login forms, custody, and tracking records Sample storage records Sample preparation logs Standard traceability logs Equipment calibration logs Sample analysis run logs Equipment maintenance, testing, and inspection logs Corrective action forms Reported field sample results Reported results for standards, quality control checks, and quality control samples Data completeness checklists Sample storage and disposal records Telephone logs Extraction/clean-up records Raw data EDD	Tt Project File, long-term data package storage at third-party professional document storage firm. The EDDs and corresponding databases will be formatted in both Navy and MEDEP formats.
Data Assessment Documents and Records Field Sampling Audit Checklist (if an audit is conducted) Analytical Audit Checklist (if an audit is conducted) Data Validation Memoranda	Tt Project File. Data validation reports will also be included in the field investigation data package.

Document / Record	Where Maintained
Other Documents HASP All versions of SAP Field investigation data package All versions of project reports (e.g., RI, FS)	Tt Project File

SAP Worksheet #30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory/ Organization(1)	Backup Laboratory/ Organization
Soil	Metals – Copper, Lead	See Worksheet #18	See Worksheet #18	21 calendar days	Mitkem Laboratories	Not applicable
	PCB (Homologs)	See Worksheet #18	See Worksheet #18	21 calendar days	Mitkem Laboratories	Not applicable

SAP Worksheet #31 – Planned Projects Assessment Table

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Laboratory System Audit(1)	Every 2 years	External	DoD ELAP	DoD ELAP accrediting body	Laboratory QAM or Laboratory Manager	Laboratory QAM or Laboratory Manager	Laboratory QAM or Laboratory Manager

1. Mitkem Laboratories is DoD ELAP accredited. The DoD ELAP accreditation letter is included in Appendix C.

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

(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Time Frame of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Time Frame for Response
Laboratory System Audit	Written audit report	Edward Lawler, Deputy Director for Quality Services, Mitkem Laboratories	Specified by DoD ELAP accrediting body	Letter	DoD ELAP Accrediting body	Specified by DoD ELAP accrediting body

SAP Worksheet #33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data validation report	Per SDG	After validation is complete	Tt DVM or designee	Tt PM, Project File
Major analysis problem identification (internal memorandum)	When persistent analysis problems are detected	Immediately	Tt QAM	Tt PM, Tt QAM, Tt Program Manager, Project File
Project monthly progress report	Monthly	Monthly	Tt PM	Tt PM
Field progress report	Daily, oral, during the course of sampling	Every day that field sampling occurs	Tt FOL	Tt PM
Laboratory QA report	When significant plan deviations result from unanticipated circumstances	Immediately	Subcontracted laboratory	Tt, Project File

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

(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The Tt FOL or designee will review and sign each chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and that the sample information is accurate. The form will be signed by the sampler, and a copy will be retained for the Tt Project File, and the Tt PM. See Tt SOP SA-6.3.	Internal	Tt sampler and FOL
SAP Sample Tables	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	Tt FOL or designee
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	Tt FOL or designee
Sample Coordinates	Verify that sample locations are correct and in accordance with the SAP proposed locations.	Internal	Tt FOL or designee
Field SOPs/Field Logs/Sample Collection	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the SAP was implemented and carried out as written and that any deviations are documented.	Internal	Tt TOM or designee
Analytical SOPs	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Internal	Laboratory QAM, Mitkem, Inc.
Documentation of Method QC results	Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the laboratory will contact Tt for guidance prior to report preparation.	Internal	Laboratory QAM, Mitkem, Inc.
Field QC Samples	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	Tt FOL or designee
Chain-of-Custody Forms	The laboratory sample custodian will review the sample shipment for completeness and integrity and will sign accepting the shipment. The Tt data validator will check that each chain-of-custody Form was signed/dated by the Tt FOL or designee relinquishing the samples and also by the laboratory sample custodian receiving the samples for analyses.	Internal/ External	Laboratory sample custodian/Tt data validator

Verification Input	**Description**	**Internal / External**	**Responsible for Verification** (name, organization)
Analytical Data Package	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory QAM will sign the case narrative for each data package.	Internal	Laboratory QAM
Analytical Data Package	The data package will be verified for completeness by the Tt data validator. Missing information will be requested from the laboratory.	External	Tt data validator
EDDs	The electronic data will be verified against the chain-of-custody and hard copy data packages for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the method detection limit and reporting limit will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifier.	External	Tt data validator

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

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

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Chain-of-Custody Form	Ensure that the custody and integrity of the samples was maintained from collection to analysis and that the custody records are complete and any deviations are recorded.	Tt project chemist or data validators
IIa	Holding Times	Review that samples were shipped and stored at the required temperatures and that sample pHs for pH-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Tt project chemist or data validators
IIa/IIb	Laboratory Data Results for Accuracy	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Tt project chemist or data validators
IIa/IIb	Laboratory Duplicate Analyses for Precision	Check the laboratory precision by reviewing the RPD or %D values from laboratory duplicate analyses, MS/MSDs, and LCS/LCSDs. Ensure compliance with the methods and project MPC accuracy goals listed in Worksheet #28.	Tt project chemist or data validators
IIa/IIb	PALs	Discuss the impact of matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant on the other target compounds reported as not detected. Document this usability issue and inform the Tt PM.	Tt project chemist or data validators
IIa/IIb	Data Validation Report	Summarize deviations from methods, procedures, or contracts. Qualify results based on method or QC deviation, and explain all the data qualifications. Print a copy of the project database depicting data qualifiers and data qualifiers codes that summarize the reasons for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	Tt project chemist or data validators
IIa, IIb	SAP QC Sample Documentation	Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. If any significant QC deviations occur, the laboratory shall have contacted the Tt PM.	Tt PM or designee

Step IIa / IIb[1]	**Validation Input**	**Description**	**Responsible for Validation** (name, organization)
IIa, IIb	Documentation of Analytical Reports for Completeness	Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final Structured Query Language (SQL) database.	Tt Project Chemist or Data Validators
IIa/IIb	PALs	Review and add PALs to the laboratory EDD. Flag samples and notify PM of samples that exceed PALs, as listed in Worksheet #15.	Tt PM or designee
IIb	QLs for Sensitivity	Ensure that the MDLs listed in Worksheet #15 were achieved.	Tt Project Chemist or Data Validators
IIb	Analytical Data Deviations	Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results.	Tt Project Chemist or Data Validators

1 IIa = Compliance with methods, procedures, and contracts (see Table 10, page 117, UFP-QAPP Manual, V.1, March 2005).
IIb = Comparison with measurement performance criteria in the SAP (see Table 11, page 118, UFP-QAPP Manual, V.1, March 2005).

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

(UFP-QAPP Manual Section 5.2.2.1)

Step IIa/IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Soil	PCB Homologs	Tier III data validation. Criteria for U.S. EPA Method 680 listed in Worksheet #s 12 and 28. If not included in Worksheet #s 12 or 28, default to QA and QC criteria provided in U.S. EPA Method 680 (U.S. EPA, November 1985), then to Region 1 EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, 12/96 and 02/04 as relevant. If not included in the worksheets or Region 1 guidelines listed above, default to DoD QSM for Environmental Laboratories, April 2009, as relevant.	DVM or designees, Tt
IIa and IIb	Soil	Select Metals – Copper and Lead	Tier III data validation. Criteria for SW-846 6010B/6020/7471A listed in Worksheet #s12 and 28. If not included in Worksheet #s 12 or 28, default to Region 1 EPA-NE Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, Part VI, November 2008. If not included in the worksheets or Region 1 guidelines listed above, default to the DoD QSM for Environmental Laboratories, April 2009.	DVM or designees, Tt

SAP Worksheet #37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment
The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the assessors determine that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics: **Completeness** o For each matrix that was scheduled to be sampled, the FOL (or other person designated by the Tt PM) acting on behalf of the Project Team will prepare a table listing planned samples/analyses compared to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified, the Tt PM and risk assessor will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tt PM will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions. **Precision** o The Project Chemist acting on behalf of the Project Team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheet #s 12 and 28. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report. **Accuracy** o The Project Chemist acting on behalf of the Project Team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCSs, LCSDs, MSs, MSDs, 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, MSs, and LCSs. 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 Tt PM and acting on behalf of the Project Team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with the SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

Comparability

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

Sensitivity

- The Project Chemist acting on behalf of the Project Team will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Project Chemist will enlist the help of the project risk assessor to evaluate deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

- The Tt PM and designated team members will evaluate whether project assumptions are valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested.

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

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

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

Identify the personnel responsible for performing the usability assessment:

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

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

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

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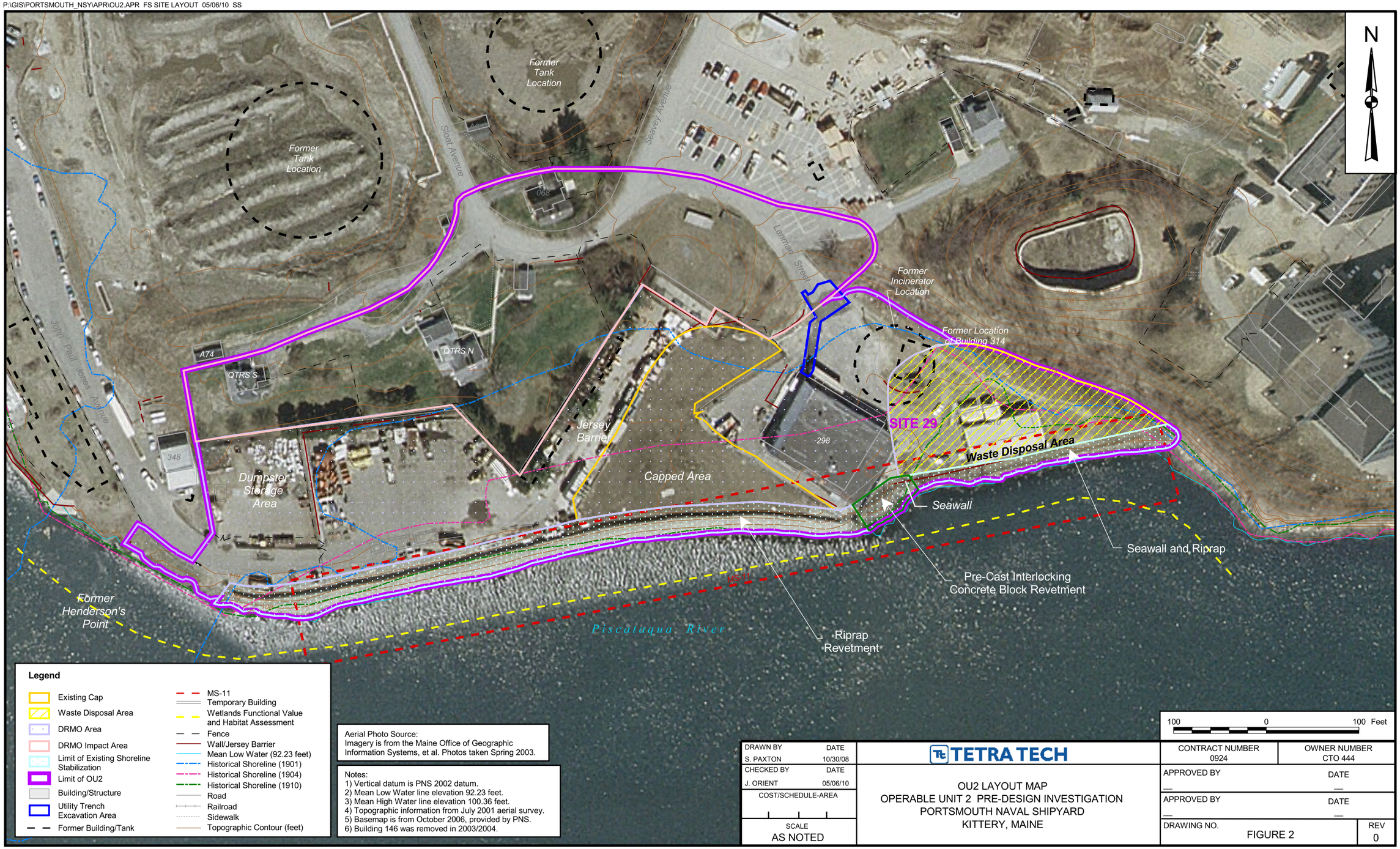
DRAWN BY T. WHEATON	DATE 04/27/10	TETRA TECH	CONTRACT NUMBER CTO 123
CHECKED BY M. KRAUS	DATE 04/30/10	PORTSMOUTH NAVAL SHIPYARD VICINITY MAP PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY ___ DATE ___
COST/SCHEDULE AREA			APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 1 / REV 0

P:\GIS\PORTSMOUTH_NSY\APR\OU2.APR FS SITE LAYOUT 05/06/10 SS



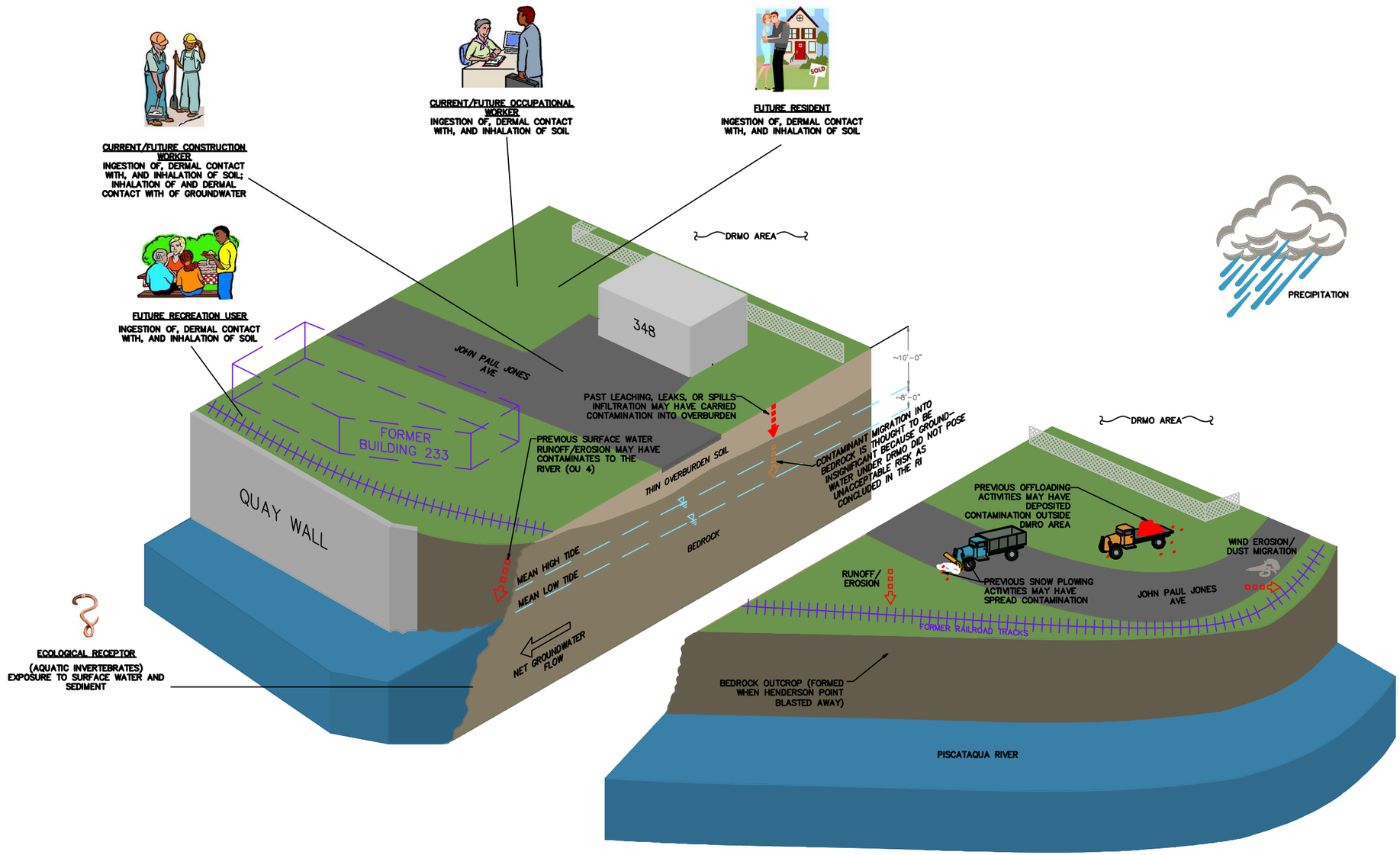
Aerial Photo Source:
Imagery is from the Maine Office of Geographic Information Systems, et al. Photos taken Spring 2003.

Notes:
1) Vertical datum is PNS 2002 datum.
2) Mean Low Water line elevation 92.23 feet.
3) Mean High Water line elevation 100.36 feet.
4) Topographic information from July 2001 aerial survey.
5) Basemap is from October 2006, provided by PNS.
6) Building 146 was removed in 2003/2004.

DRAWN BY S. PAXTON	DATE 10/30/08	TETRA TECH	CONTRACT NUMBER 0924 / OWNER NUMBER CTO 444
CHECKED BY J. ORIENT	DATE 05/06/10	OU2 LAYOUT MAP OPERABLE UNIT 2 PRE-DESIGN INVESTIGATION PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY — DATE —
COST/SCHEDULE-AREA			APPROVED BY — DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 2 / REV 0

P:\GIS\PORTSMOUTH_NSY\MAPDOCS\MXD\OU2_EXCAVATION_LOCS.MXD 05/06/10 SS

DRAWN BY J. ENGLISH	DATE 11/20/09	TETRA TECH	CONTRACT NUMBER ___
CHECKED BY M. KRAUS	DATE 04/27/10	AREA WEST OF OU 2 OPERABLE UNIT 2 PRE-DESIGN INVESTIGATION PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY ___ DATE ___
REVISED BY S. STROZ	DATE 05/06/10		APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 3 / REV 0



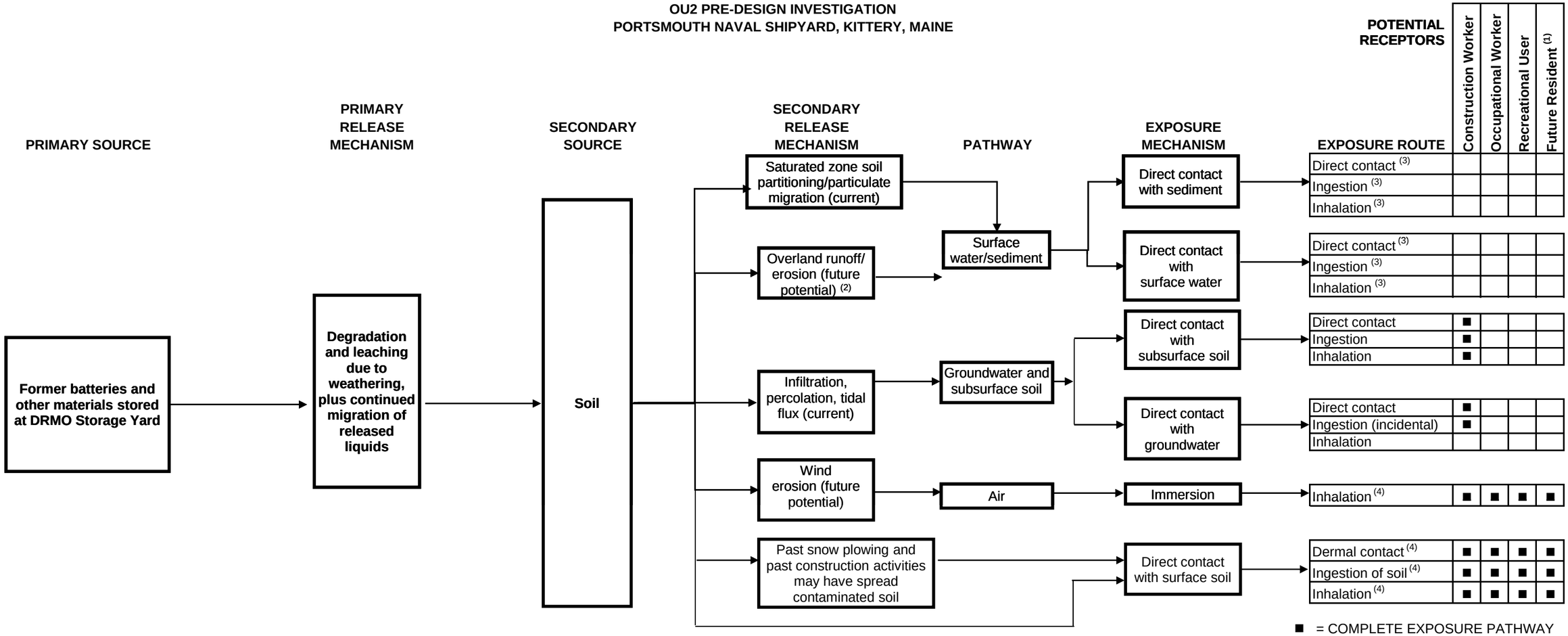
LEGEND

- CONTAMINANT MIGRATION PATHWAY
- INSIGNIFICANT MIGRATION PATHWAY
- PREVIOUS CONTAMINATION PATHWAY (FUTURE POTENTIAL)
- PREVIOUS CONTAMINATION SOURCE

DRAWN BY CK DATE 11/24/09	NAVFAC	3D CONCEPTUAL SITE MODEL AREA WEST OF OU2 OPERABLE UNIT 2 PRE-DESIGN INVESTIGATION PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	CONTRACT NO. 2229
CHECKED BY DATE			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 4A / REV. 0

R:\2229\2229C002.dwg PIT CARLY.KRAMER 5/5/2010 2:39:07 PM

FIGURE 4b
HUMAN HEALTH CONCEPTUAL SITE MODEL
OU2 PRE-DESIGN INVESTIGATION
PORTSMOUTH NAVAL SHIPYARD, KITTERY, MAINE



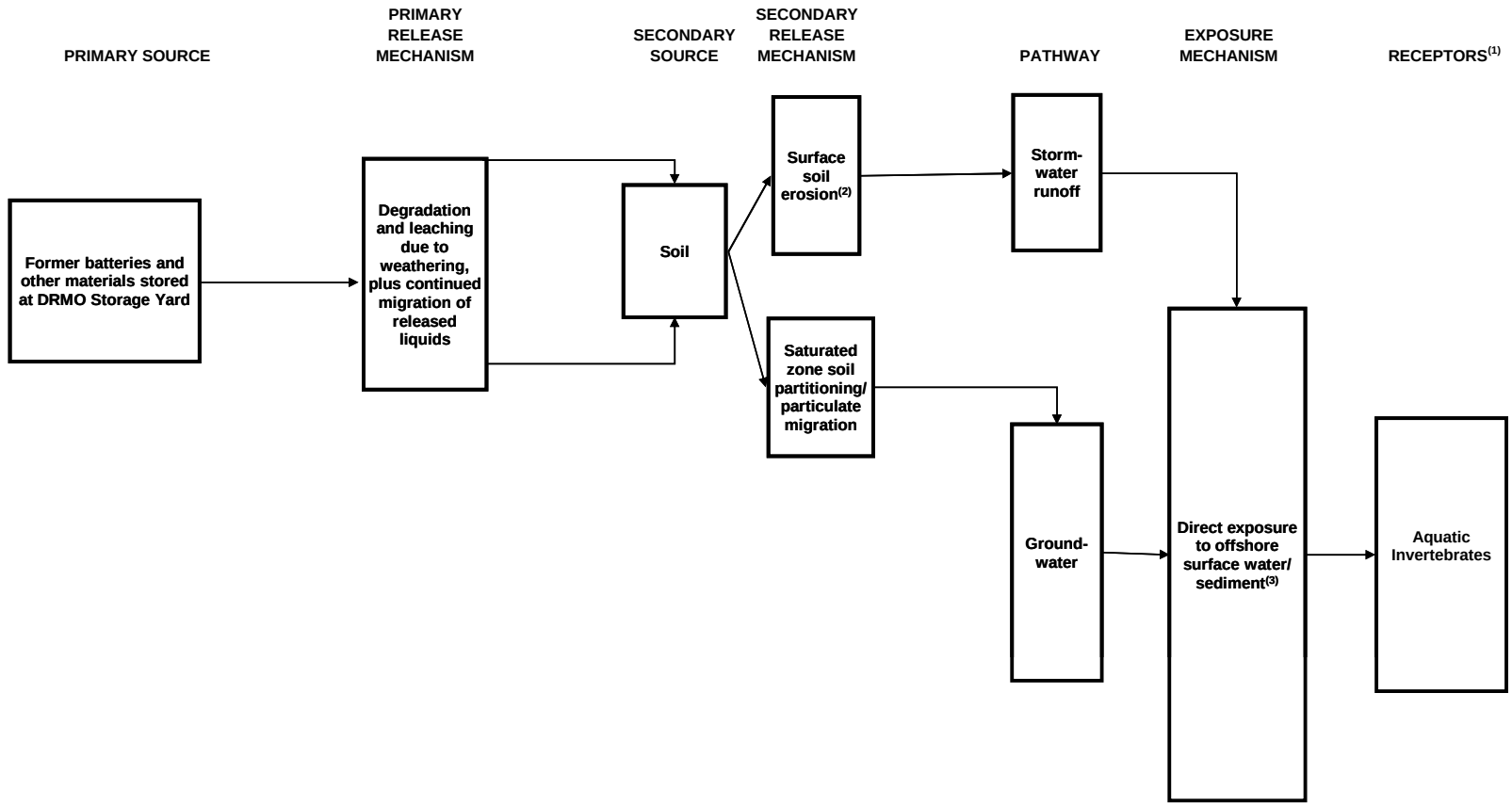
1 Groundwater exposure for a future resident is an incomplete pathway because groundwater at the site is not potable due to salinity.

2 Overland erosion was a past release mechanism until 1993 when interim measures were implemented. This is considered a future potential pathway if surface cover materials (asphalt, buildings, vegetation, or gravel) are removed and contaminated soil is exposed.

3 Human exposure to surface water or sediment is considered an incomplete pathway because there is no public access to the offshore from OU2. The shoreline is rocky and very steep, and consequently, typical recreational activities would not be possible.

4 Soil is covered with asphalt, buildings, vegetation, and/or gravel; therefore, direct contact with surface soil by receptors other than construction workers or erosion to the offshore are future concerns only if surface cover materials are disturbed.

FIGURE 4c
ECOLOGICAL CONCEPTUAL SITE MODEL
OU2 PRE-DESIGN INVESTIGATION
PORTSMOUTH NAVAL SHIPYARD, KITTERY, MAINE



1 Sufficient habitat onshore at OU2 is not available for ecological receptors; therefore, onsite ecological exposure is not considered significant for OU2.
2 As of June 2006, shoreline controls are present along the entire OU2 shoreline, and surficial accumulated debris in the eastern portion of OU2 was removed and gravel was placed over the area; therefore, erosion is no longer considered a current concern. Surface soil erosion is only of future concern if the asphalt, gravel/rip-rap, or grass-covered surfaces are distrubed and underlying soil is exposed. Plowing of snow removed from

3 There is very little sediment in the offshore area of OU2.

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DRAWN BY J. ENGLISH	DATE 12/14/09	TETRA TECH	CONTRACT NUMBER ___
CHECKED BY M. KRAUS	DATE 05/07/10	PROPOSED SAMPLING LOCATIONS OPERABLE UNIT 2 PRE-DESIGN INVESTIGATION PORTSMOUTH NAVAL SHIPYARD KITTERY, MAINE	APPROVED BY ___ DATE ___
REVISED BY ___	DATE ___		APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 5 / REV 0

APPENDIX A

FIELD DOCUMENTATION FORMS

Tetra Tech NUS, Inc.

CONTAINER SAMPLE & INSPECTION SHEET

Page_____ of _____

Project Site Name: _____
Project Number: _____
Site Identification: _____
Container Number(s): _____
Sample Type: [] Grab
[] Composite

Sample ID No. _____
Sampled By: _____
C.O.C. No.: _____
Concentration: [] High
[] Medium
[] Low

CONTAINER SOURCE

DRUM:
[] Bung Top
[] Lever Lock
[] Bolted Ring
[] Other _____

TANK:
[] Plastic
[] Metal
[] Other _____

OTHER: _____

CONTAINER DESCRIPTION

COLOR: _____

CONDITION: _____

MARKINGS: _____

VOL. OF CONTENTS: _____

OTHER: _____

CONTAINER DISPOSITION

SAMPLED: _____

OPENED BUT NOT SAMPLED:
Reason _____
_____

NOT OPENED:
Reason _____
_____

CONTENTS DESCRIPTION

SINGLE PHASED: _____
_____

MULTIPHASE :

	Layer 1	Layer 2	Layer 3
Phase (Sol. or Liq.)	_____	_____	_____
Color	_____	_____	_____
Viscosity	L, M or H	L, M or H	L, M orH
% of Total Volume	_____	_____	_____

MONITOR READING:

SAMPLE and /or INSPECTION DATE & TIME:
_____HRS.
METHOD:_____

SAMPLER(S) and / or INSPECTOR(S) SIGNATURE:

ANALYSIS:

Tetra Tech NUS, Inc.

PROJECT: _____ **JOB #:** _____
LOCATION: _____ **DATE:** _____
PROJECT MANAGER: _____ **FOL:** _____

DAILY ACTIVITIES CHECKLIST

Startup Checklist

Activity	Yes	No	N/A
Pertinent site activities/information entered into site logbook			
All onsite personnel listed in logbook			
Required medical information onsite for all workers (TtNUS and Subcontractors)			
Required MSDS's onsite			
Proper equipment calibrations performed (list equipment)			
1 _____			
2 _____			
3 _____			
4 _____			
Calibration logs filled out			
Tailgate H&S meeting held prior to beginning field activities			
Required work permits filled out/signed			
Required utility clearances obtained			
Required PPE onsite and in use			
Information required to be posted is in place			
(OSHA poster, hospital route, key phone numbers, etc.)			

Exit Checklist

Activity	Yes	No	N/A
Logbooks completely and comprehensively filled out			
Field forms complete and accounted for/properly filed			
Samples properly packaged/shipped			
COCs faxed to appropriate in-house personnel			
All equipment accounted for, on charge if needed, and properly secured			
All personnel accounted for			
Arrangements made for upcoming work (permits, clearances, equipment, etc.)			
Site properly secured			

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

DAILY ACTIVITIES RECORD

PROJECT NAME: _____ **PROJECT NUMBER:** _____

CLIENT: _____ **LOCATION:** _____

DATE: _____ **ARRIVAL TIME:** _____

Tt NUS PERSONNEL: _____ **DEPARTURE TIME:** _____

CONTRACTOR: _____ **DRILLER:** _____

ITEM	QUANTITY ESTIMATE	QUANTITY TODAY	PREVIOUS TOTAL QUANTITY	CUMULATIVE QUANTITY TO DATE

COMMENTS: _____

_____

_____

_____

APPROVED BY:

_____ _____

Tt NUS REPRESENTATIVE DRILLER

DATE: _____

TETRA TECH NUS
FIELD TASK MODIFICATION REQUEST FORM

Project/Installation Name _____ CTO & Project Number _____ Task Mod. Number _____

Modification To (e.g. Work Plan) _____ Site/Sample Location _____ Date _____

Activity Description: _____

Reason for Change: _____

Recommended Disposition: _____

Field Operations Leader (Signature) _____ Date _____

Approved Disposition:

Project/Task Order Manager (Signature) _____ Date _____

Distribution:

Program/Project File –
Project/Task Order Manager –
Field Operations Leader –

Other: _____

Tetra Tech NUS, Inc.

PROJECT: _____ LOCATION: _____

JOB & CTO #: _____ MOBILIZATION DATE: _____

PROJECT MANAGER: _____ RETURN DATE: _____

FIELD PROJECT PRE-MOBILIZATION CHECKLIST

TRAVEL

___ Airline reservations
___ Hotel reservations/BOQs
___ Vehicle rental
___ Itinerary
___ Phone/pager number

DRILLING/DPT/SURVEY

Subcontractor

___ POC phone #/address
___ Drill Specification RFP
___ Contact (time & place to meet)
___ Confirm subcontract w/ TtNUS Procurement
___ Health and Safety documentation for all personnel on site
___ Copy of Drillers license
___ Well / boring permits

Utilities (2 weeks lead time)

___ Contact Site POC (Date:_____)
___ Contact Local "Call Before You Dig"
___ Utility Clearance Form

Forms

___ Boring logs / Test Pit logs
___ Well construction / development forms
___ Daily activity forms
___ IDW inventory
___ IDW drum labels
___ Chemical Inventory
___ MSDS's

EQUIPMENT MOBILIZATION

___ Equipment Requisition form completed / equipment ordered
___ 3rd Party rental / misc. equipment ordered
___ Equipment calibration forms
___ Span / calibration gas and regulator

SAMPLING

Forms

___ Sample log sheets
___ Low-flow purge data sheets
___ COC records
___ COC seals
___ Sample labels (from database group)

Laboratory

___ POC address/phone#
___ Order bottles / preservatives
___ Shipping address, also check Sat. address
___ Bottle & preservation req'ts from lab
___ _____

MISCELLANEOUS

Schedule

___ Plan field operations w/ Project manager

Documents for Field Program

___ Logbook(s)
___ Field Sampling plan
___ Health & Safety plan
___ Maps
___ H & S Guidance Manual

Authorization

___ Kick-off meeting held
___ Gov't rate letter
___ H&S/OSHA 40-hour certifcate
___ 8-Hour Refresher Training Certificate
___ Medical Clearance Letter
___ Supervisory Training Certificate
___ Health & Safety Clearance Letter
___ Full-size OSHA Poster

HYDROGEOLOGY EQUIPMENT

___ Slug test/pumping test forms
___ Groundwater elevation data sheets
___ Graph paper
___ Data Logger/transducer/data cable
___ Existing well construction & water level data
___ M-Scope, slug

SHIPPING

Forms

___ FedEx Airbills, local dropoff location & hours
___ FedEx Gov. Acct# (1771-8058-0)
___ Lab Shipping Labels
___ Warehouse Shipping Labels
___ Blank Labels

Supplies

___ Tape
___ Packing materials
___ Baggies, Large garbage bags

OTHER

___ Site POC name/phone #
___ Personnel information to POC
___ Mobilization schedule to POC
___ Site access authorizations
___ Field office / trailer arrangements made
___ Electric, phone hookups arranged
___ Steel-toed boots, safety glasses, & hard hat
___ First aid equipment
___ Insect repellent
___ _____
___ _____

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Project Site Name: _____ Sample ID Number: _____
Project Number: _____ Sampled By: _____
Sample Location: _____ C.O.C. Number: _____
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other _____

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: _____
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:

[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
Volatiles	Cool 4°C & HCl		YES / NO
Semivolatiles	Cool 4°C		YES / NO
Pesticide / PCB	Cool 4°C		YES / NO
Metals	Cool 4°C & HNO_3		YES / NO
Cyanide	Cool 4°C & NaOH		YES / NO

OBSERVATIONS / NOTES:

Signature(s):

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

APPENDIX B

FIELD STANDARD OPERATING PROCEDURES

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6.0 DEVIATIONS **6**

1.0 PURPOSE

The purpose of this document is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix.
- Sorting of data by depth.
- Maintenance of consistency (field, laboratory, and data base sample numbers).
- Accommodation of all project-specific requirements.
- Accommodation of laboratory sample number length constraints (maximum of 20 characters).

2.0 SCOPE

The methods described in this procedure shall be used consistently for all projects requiring electronic data.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Program Manager - It shall be the responsibility of the Program Manager (or designee) to inform contract-specific Project Managers of the existence and requirements of this Standard Operating Procedure.

Project Manager - It shall be the responsibility of the Project Manager to determine the applicability of this Standard Operating Procedure based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the sample nomenclature is thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and is consistent with this Standard Operating Procedure if relevant. It shall be the responsibility of the project manager to ensure that the Field Operations Leader is familiar with the sample nomenclature system.

Field Operations Leader - It shall be the responsibility of the Field Operations Leader to ensure that all field technicians or sampling personnel are thoroughly familiar with this Standard Operating Procedure and the project-specific sample nomenclature system. It shall be the responsibility of the Field Operations Leader to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

5.0 PROCEDURES

5.1 Introduction

The sample identification (ID) system can consist of as few as 8 but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the lab has three segments and shall be as follows where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

(1) Soil and Sediment Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NNNN 4-Characters
Site Identifier	Sample Type	Sample Location	Sample Depth

(2) Aqueous (groundwater or surface water) Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NN 2-Characters	-A
Site Identifier	Sample type	Sample Location	Round Number	Filtered Sample only

(3) Biota Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	AA 2-Characters	NNN 3-Characters
Site Identifier	Sample Type	Sample Location	Species Identifier	Sample Group Number

5.2 Sample Identification Field Requirements

The various fields in the sample ID will include but are not limited to the following:

- Site Identifier
- Sample Type
- Sample Location
- Sample Depth
- Sampling Round Number
- Filtered
- Species Identifier
- Sample Group Number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary since many facilities/sites have multiple individual sites, SWMUs, operable units, etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six-characters (alpha, numeric, or a mixture). The six-characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to

three-characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

A two-digit round number will be used to track the number of aqueous samples taken from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001 and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

Site Identifier - Examples of site numbers/designations are as follows:

A01 - Area of Concern Number 1
125 - Solid Waste Management Unit Number 125
000 - Base or Facility Wide Sample (e.g., upgradient well)
BBG - Base Background

The examples cited are only suggestions. Each Project Manager (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample Type - Examples of sample types are as follows:

AH - Ash Sample
AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample

SG - Soil Gas Sample
SL - Sludge Sample
SP - Seep Sample
SS - Surface Soil Sample
ST - Storm Sewer Water Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WP - Wipe Sample
WS - Waste/Solid Sample
WW - Wastewater Sample

Sample Location - Examples of the location field are as follows:

001 - Monitoring Well 1
N32E92 - Grid location 32 North and 92 East
D096 - Investigation derived waste drum number 96

Species Identifier - Examples of species identifier are as follows:

BC - Blue Crab
GB - Blue Gill
CO - Corn
SB - Soybean

5.4 Examples of Sample Nomenclature

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full body analysis the first time a minnow trap was checked at grid location A25 of SWMU 1415 three small blue gills were captured, collected and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415) the sample ID designation given was 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash "-F".

5.5 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature)

Field QA/QC will be designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC Type	Date	Sequence Number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
WB = Source Water Blank

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log sheet (see SOP on Field Documentation).

5.6 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day for a filtered ground water sample collected on June 3, 2000 would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000 would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001 would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site specific planning documents.

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		Effective Date 06/99	Revision 1
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject BOREHOLE AND SAMPLE LOGGING		Approved D. Senovich	

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1.0 PURPOSE

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

2.0 SCOPE

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

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

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

5.0 PROCEDURES

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

5.1 Materials Needed

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

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

5.2 Classification of Soils

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

FIGURE 1
BORING LOG (EXAMPLE)

BORING LOG

Page ___ of ___

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

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

* When rock coring, enter rock brokeness.
** Include monitor reading in 5 foot intervals @ borehole. Increase reading frequency if elevated response-read.

Drilling Area Background (ppm): ☐

Remarks: _____

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

FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)

COARSE-GRAINED SOILS More Than Half of Material is LARGER Than No. 200 Sieve Size				
FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES
GRAVELS (50% or More of Coarse Fraction Is Larger Than No. 4 Sieve Size)	CLEAN GRAVELS (Less Than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well graded gravels, gravel-sand mixtures, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines.
	GRAVELS WITH FINES (More Than 12% Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.
SANDS (50% or More of Coarse Fraction Is Smaller Than No. 4 Sieve Size)	CLEAN SANDS (Less Than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sand, gravelly sands, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.
	SANDS WITH FINES (More Than 12% Fines)	Non-plastic fines (for identification procedures, see MCL)	SM	Silty sands, poorly graded sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.

FINE-GRAINED SOILS More Than Half of Material is SMALLER Than No. 200 Sieve Size							
FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)						GROUP SYMBOL	TYPICAL NAMES
		Identification Procedures on Fraction Smaller Than No. 40 Sieve Size					
		DRY STRENGTH (Crushing Characteristics)	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)			
SILTS AND CLAYS Liquid Limit <50		None to Slight	Quick to Slow	None		ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
		Medium to High	None to Very Slow	Medium		CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
		Slight to Medium	Slow	Slight		OL	Organic silts and organic silt-clays of low plasticity.
SILTS AND CLAYS Liquid Limit >50		Slight to Medium	Slow to None	Slight to Medium		MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
		High to Very High	None	High		CH	Inorganic clays of high plasticity, fat clays.
		Medium to High	None to Very Slow	Slight to Medium		OH	Organic clays of medium to high plasticity.
HIGHLY ORGANIC SOILS		Readily identified by color, odor, spongy feel and frequently by fibrous texture.				Pt	Peat and other organic soils

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

DENSITY OF GRANULAR SOILS

DESIGNATION	STANDARD PENETRATION RESISTANCE* BLOWS/FOOT
Very Loose	0-4
Loose	5-9
Medium Loose	10-29
Dense	30-50
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS

CONSISTENCY	UNCONFINED COMPRESSIVE STRENGTH (TONS/SQ. FT.)	STANDARD PENETRATION RESISTANCE* BLOWS/FOOT	FIELD IDENTIFICATION METHOD
Very Soft	Less than 0.25	0 to 2	Easily penetrated several inches by fist
Soft	0.25 to 0.50	2 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.50 to 1.0	4 to 8	Can be penetrated several inches by thumb.
Stiff	1.0 to 2.0	8 to 16	Readily indented by thumb.
Very Stiff	2.0 to 4.0	16 to 32	Readily indented by thumbnail.
Hard	More than 4.0	Over 32	Indented with difficulty by thumbnail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)		
Descriptive Terms	Scratcher or Knife Effects	Hammer Effects
Soft	Easily Gouged	Crushes when pressed with hammer
Medium Soft	Can be Gouged	Breaks (one blow); crumbly edges
Medium Hard	Can be Scratched	Breaks (one blow); sharp edges
Hard	Cannot be Scratched	Breaks conchoidally (several blows); sharp edges

ROCK BROKENNESS		
Descriptive Terms	Abbreviation	Spacing
Very Broken	(V. Br.)	< 2"
Broken	(Br.)	2"-7"
Blocky	(Bl.)	7-12"
Massive	(M.)	> 12"

LEGEND :

SOIL SAMPLER - TYPES

3" Split Spoon Sample

STS 3" O.D. Undisturbed Sample

O - Other Samples, Specify in Remarks

ROCK SAMPLER - TYPES

X-NX (Conventional) Core (2.1-13" O.D.)

O-NQ (Diamond) Core (2.1-1.9" O.D.)

Z - Other Core Sizes, Specify in Remarks

WATER LEVELS

▽ 9.0' Initial Level with Date & Depth

▽ 9.0' Stabilized Level with Date & Depth

5.2.1 USCS Classification

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

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

FIGURE 2

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

5.2.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

FIGURE 3

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ($CaCO_3$). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

FIGURE 4

GRAIN SIZE CLASSIFICATION FOR ROCKS

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).
- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.
- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.
- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the works "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

$$RQD\ \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C - Coarse	Lt - Light	Yl - Yellow
Med - Medium	BR - Broken	Or - Orange
F - Fine	BL - Blocky	SS - Sandstone
V - Very	M - Massive	Sh - Shale
Sl - Slight	Br - Brown	LS - Limestone
Occ - Occasional	Bl - Black	Fgr - Fine-grained
Tr - Trace		

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.

- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.

- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.

- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.

- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5
COMPLETED BORING LOG (EXAMPLE)

BORING LOG

Page 1 of 1

PROJECT NAME: NSB- SITE
PROJECT NUMBER: 9594
DRILLING COMPANY: SOILTEST CO.
DRILLING RIG: CME-55
BORING NUMBER: SB/MW 1
DATE: 3/8/96
GEOLOGIST: SJ CONTI
DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/FL) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS*	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**
S-1 @ 0800	0.0	7/6	1.5/2.0		M DENSE	BRN TO BLK	SILTY SAND - SOME	SM	MOIST SL. ORG. ODOR	5	0	0	0
	2.0	9/10					ROCK FR - TR BRICKS (FILL)		FILL TO 4'±				
	4.0			4.0									
S-2 @ 0810	6.0	5/7 9/8	2.9/2.0	7'± ▽	M DENSE	BRN	SILTY SAND - TR FINE GRAVEL	SM	MOIST - W ODOR NAT. MATL. TOOK SAMPLE SB01-0406 FOR ANALYSIS	10	0	-	-
	8.0			8.0									
S-3 @ 0820	10.0	6/8 17/16	1.9/2.0		DENSE	TAN BRN	FINE TO COARSE SAND TR. F. GRAVEL	SW	WET HIT WATER ≈ 7'±	0	0	0	0
	12.0			12.0									
S-4 @ 0830	14.0	7/6 5/8	1.6/2.0		STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	.5	-	-
	15.0			15.0					AUGER REF @ 15'				
0/5	①		4.0/5.0	11 BLW 16 / 19'	M HARD	BRN	SILTSTONE	VER	WEATHERED LO % JNTS @ 15.5 WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
	20.0				HARD	GRAY	SANDSTONE - SOME SILTSTONE	BR	LOSING SOME DRILL H2O @ 17'± SET TEMP 6" CAS TO 15.5				
4.0/5.0	②		5.0/5.0						SET 2" ⌀ PVC SCREEN 16-25 SAND 14-25 PELLETS 12-14	0	0	0	0
	25.0			25									

* When rock coring, enter rock brokeness.
** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

• 1-20Z 1-80Z

Drilling Area Background (ppm): 0

Remarks: CME-55 R16, 4¼" ID HSA - 9" OD ±
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP
NX CORE IN BEDROCK RUN ① = 25 min, RUN ② = 15 min

Converted to Well: Yes ✓ No _____ Well I.D. #: MW-1

- Enter color of the material in the appropriate column.

- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:

  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent

- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.

- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.

- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:

  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.

  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.

  - Particle shape - flat, elongated, or flat and elongated.

  - Maximum particle size or dimension.

  - Water level observations.

  - Reaction with HCl - none, weak, or strong.

- Additional comments:

  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.

  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.

  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.

  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.

- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.

- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.

- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.

- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.

- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:

  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.

- Remarks at the bottom of Boring Log shall include:

  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.

- As a final check the boring log shall include the following:

  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.

  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.3 ~~Classification of Soil and Rock from Drill Cuttings~~

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.

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1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.

2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scares and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.

4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.

5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain

6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.

7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.

8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly though conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

Nominal Voltage	Minimum Clearance
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.

2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

   Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.

4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
OSHA 29 CFR 1926(b)(2)
OSHA 29 CFR 1926(b)(3)
TtNUS Utility Locating and Clearance Policy
TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

ONE-CALL SYSTEMS INTERNATIONAL CONDENSED DIRECTORY

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5348

Arkansas
Arkansas One Call System, Inc.
1-800-482-8998

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-922-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8989

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007

Louisiana
Louisiana One Call System, Inc.
1-800-272-3020

Maine
Dig Safe System, Inc.
1-888-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-888-344-7233

Michigan
Miss Dig System, Inc.
1-800-482-7171

Minnesota
Gopher State One Call
1-800-252-1166

Mississippi
Mississippi One-Call System, Inc
1-800-227-6477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344

Nebraska
Diggers Hotline of Nebraska
1-800-331-5666

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-888-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400

New York
Dig Safely New York
1-800-962-7962
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-632-4949

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0988

Oklahoma
Call Okie
1-800-522-6543

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-888-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111

ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah
Blue Stakes of Utah
1-800-662-4111

Vermont
Dig Safe System, Inc.
1-888-344-7233

Virginia
Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington
Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating Council
509-456-8000

West Virginia
Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin
Diggers Hotline, Inc.
1-800-242-8511

Wyoming
Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia
Miss Utility
1-800-257-7777

Alberta
Alberta One-Call Corporation
1-800-242-3447

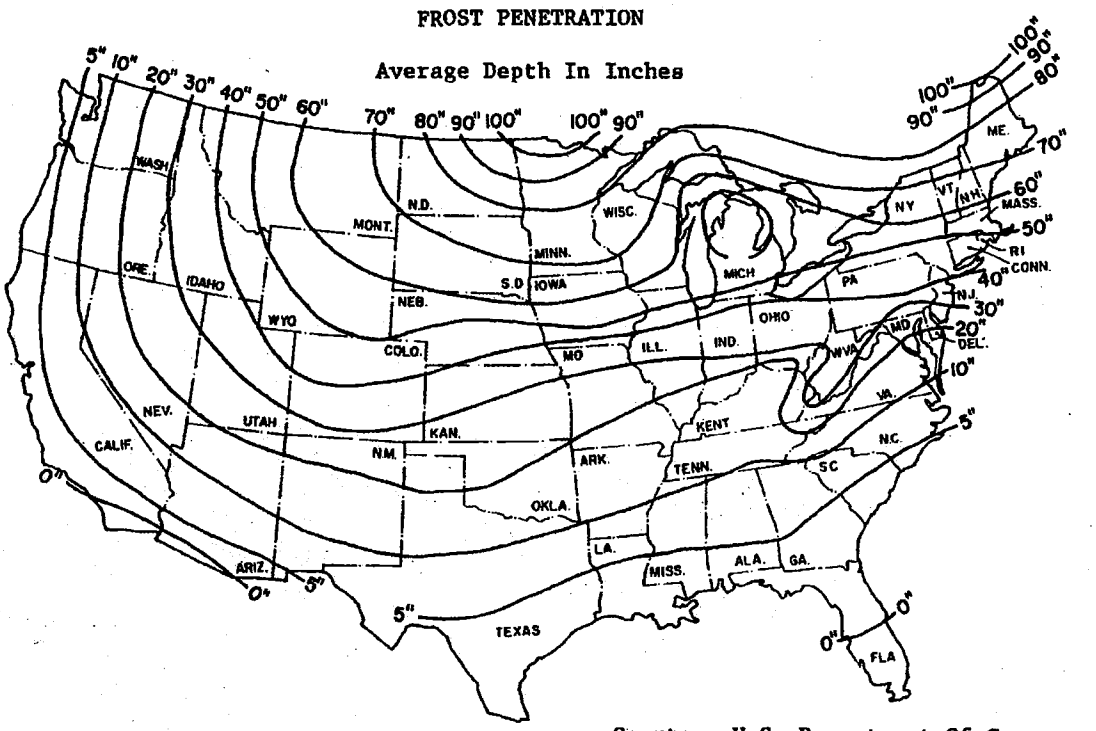
British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-400-2255

Quebec
Info-Excavation
1-800-663-9228

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

ATTACHMENT 3
UTILITY CLEARANCE FORM

Client: _____ Project Name: _____

Project No.: _____ Completed By: _____

Location Name: _____ Work Date: _____

Excavation Method/Overhead Equipment: _____

1. Underground Utilities — Circle One

a)	Review of existing maps?	yes no N/A
b)	Interview local personnel?	yes no N/A
c)	Site visit and inspection?	yes no N/A
d)	Excavation areas marked in the field?	yes no N/A
e)	Utilities located in the field?	yes no N/A
f)	Located utilities marked/added to site maps?	yes no N/A
g)	Client contact notified Name _____ Telephone: _____ Date: _____	yes no N/A
g)	State One-Call agency called? Caller: _____ Ticket Number: _____ Date: _____	yes no N/A
h)	Geophysical survey performed? Survey performed by: _____ Method: _____ Date: _____	yes no N/A
i)	Hand excavation performed (with concurrent use of utility detection device)? Completed by: _____ Total depth: _____ feet Date: _____	yes no N/A
j)	Trench/excavation probed? Probing completed by: _____ Depth/frequency: _____ Date: _____	yes no N/A

2. Overhead Utilities — Present Absent

a)	Determination of nominal voltage	yes no N/A
b)	Marked on site maps	yes no N/A
c)	Necessary to lockout/insulate/re-route	yes no N/A
d)	Document procedures used to lockout/insulate/re-route	yes no N/A
e)	Minimum acceptable clearance (SOP Section 5.2): _____	

3. Notes:

_____

_____

_____

Approval:

_____ _____

Site Manager/Field Operations Leader Date

c: PM/Project File
Program File

ATTACHMENT 4
OSHA LETTER OF INTERPRETATION

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

Question: *Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651(Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

> When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

ATTACHMENT 4 (Continued)

> When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, "acceptable means" must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either "other acceptable means" or "safe and acceptable means." The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified "careful probing or hand digging" as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language "to allow other, *equally effective means* of locating such installations." The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used *where the utilities have not been marked and detection equipment is not being used* – "probing with hand-held tools." This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

> OSHA received two comments * * * and input from ACCSH [OSHA's Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping 'such as probing with hand-held tools' from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of "acceptable means" in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a "shooter" (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an "acceptable means" for locating underground utilities.

ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA=s interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at http://www.osha.gov.

TETRA TECH	STANDARD OPERATING PROCEDURES	Number SA-1.3	Page 1 of 31
		Effective Date 04/072008	Revision 9
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SOIL SAMPLING		Approved Tom Johnston T.E. Johnston	

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1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used to collect surface, near-surface, and subsurface soil samples. Additionally, it describes the methods for sampling of test pits and trenches to determine subsurface soil and rock conditions and for recovery of small-volume or bulk samples from pits.

2.0 SCOPE

This document applies to the collection of surface, near-surface, and subsurface soil samples exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites for laboratory testing, onsite visual examination, and onsite testing.

3.0 GLOSSARY

Composite Sample - A composite sample is a combination of more than one grab sample from various locations and/or depths and times that is homogenized and treated as one sample. This type of sample is usually collected when determination of an average waste concentration for a specific area is required. Composite samples shall not be collected for volatile organics analysis.

Confined Space - As stipulated in 29 Code of Federal Regulations (CFR) 1910.146, a confined space means a space that: (1) is large enough and so configured that an employee can bodily enter and perform assigned work; (2) has limited or restricted means for entry or exit (e.g., tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations); and (3) is not designed for continuous employee occupancy. TtNUS considers all confined space as permit-required confined spaces.

Grab Sample - One sample collected at one location and at one specific time.

Hand Auger - A sampling device used to extract soil from the ground.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

Sample for Non-Volatile Analyses - Includes all chemical parameters other than volatile organics (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into resistant materials using a drive weight mounted in the drilling string. A standard split-barrel sampler is typically available in two common lengths, providing either 20-inch or 26-inch longitudinal clearance for obtaining 18-inch or 24-inch-long samples, respectively. These split-barrel samplers commonly range in size from 2 to 3.5 inches OD. The larger sizes are commonly used when a larger volume of sample material is required (see Attachment B).

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine shallow subsurface conditions for engineering, geological, and soil chemistry exploration and/or sampling purposes. These pits are excavated manually or by machine (e.g., backhoe, clamshell, trencher, excavator, or bulldozer).

Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, selecting proposed sampling locations, and selecting field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring, and excavation activities and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation, and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface, near-surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits, and trenches and for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and/or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions that are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Health and safety precautions are identified for individual sample collection procedures throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard or uneven surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas, along roadways and highways.

Methods of avoiding these hazards are provided below.

Knee injuries – If kneeling is required during soil sampling, this could result in knee injuries from stones/foreign objects and general damage due to stress on the joints. To minimize this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.
- Stretch ligaments, tendons and muscles before, during and after. Take breaks as frequently as necessary.
- Report pre-existing conditions to the SSO if you feel this activity will aggravate an existing condition.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample locations. To minimize these hazards:

- Pre-survey sampling locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations - To prevent cuts and lacerations associated with soil sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.

- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut – do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken sample jars or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel**. Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

The following procedures address surface and subsurface sampling.

CAUTION

Each situation must be evaluated individually to determine the applicability and necessity for obtaining a utility clearance ticket/dig permit. Common sense dictates, prior to digging or boring with power equipment, no matter what the depth, or digging by hand in a manner that could damage unprotected underground utilities, that a dig permit is required. See SOP HS-1.0, Utility Locating and Excavation Clearance, for additional clarification. If you do not know or are unsure as to whether a ticket is necessary – **Get the Ticket**.

6.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they migrate to the water table, and can establish the amount of contamination absorbed or adsorbed on aquifer solids that have the potential of contributing to groundwater contamination.

Soil types can vary considerably on a hazardous waste site. These variations, along with vegetation, can affect the rate of contaminant migration through the soil. It is important, therefore, that a detailed record be maintained during sampling operations, particularly noting sampling locations, depths, and such characteristics as grain size, color, and odor. Subsurface conditions are often stable on a daily basis and may demonstrate only slight seasonal variation especially with respect to temperature, available oxygen and light penetration. Changes in any of these conditions can radically alter the rate of chemical reactions or the associated microbiological community, thus further altering specific site conditions. Certain vegetation species can create degradation products that can alter contaminant concentrations in soil. This is why vegetation types and extent of degradation of this foliage must be recorded. To prevent degradation, samples must be kept at their at-depth temperature or lower, protected from direct light, sealed tightly in approved glass containers, and be analyzed as soon as possible after collection. In addition, to the extent possible, vegetation should be removed from the sample.

The physical properties of the soil, its grain size, cohesiveness, associated moisture, and such factors as depth to bedrock and water table, will limit the depth from which samples can be collected and the method required to collect them. It is the intent of this document to present the most commonly employed soil sampling methods used at hazardous waste sites.

6.2 Soil Sample Collection

6.2.1 Procedure for Preserving and Collecting Soil Samples for Volatile Organic Compound Analysis

Samples collected using traditional methods such as collection in a jar with no preservation have been known to yield non-representative samples due to loss of volatile organic compounds (VOCs). To prevent such losses, preservation of samples with methanol or sodium bisulfate may be used to minimize volatilization and biodegradation. This preservation may be performed either in the field or laboratory, depending on the sampling methodology employed. Because of the large number of sampling methods and associated equipment required, careful coordination between field and laboratory personnel is needed.

Soil samples to be preserved by the laboratory are currently being collected using Method SW-846, 5035. For samples preserved in the field, laboratories are currently performing low-level analyses (sodium bisulfate preservation) and high- to medium-level analyses (methanol preservation) depending on the needs of the end user.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

6.2.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for volatile organic analysis that are to be preserved at the laboratory shall be obtained using a hermetically sealed sample vial such as an EnCore™ sampler. Each sample shall be

obtained using a reusable sampling handle (T-handle) that can be provided with the EnCore™ sampler when requested and purchased. Collect the sample in the following manner for each EnCore™ sampler:

1. Scene Safety - Evaluate the area where sampling will occur. Ensure that the area is safe from physical, chemical, and natural hazards. Clear or barricade those hazards that have been identified.

2. Wear the appropriate personal protective equipment (PPE). This will include, at a minimum, safety glasses and nitrile surgeon's gloves. If you must kneel on the ground or place equipment on the surface being sampled, cover the ground surface with plastic to minimize surface contamination of your equipment and clothing. Wear knee pads to protect your knees from kneeling on hard or uneven surfaces.

3. Load the Encore™ sampler into the T-handle with the plunger fully depressed.

4. Expose the area to be sampled using a hand trowel or similar device to remove surface debris.

5. Press the T-handle against the freshly exposed soil surface, forcing soil into the sampler. The plunger will be forced upward as the cavity fills with soil.

6. When the sampler is full, rotate the plunger and lock it into place. If the plunger does not lock, the sampler is not full. This method ensures there is no headspace. Soft soil may require several plunges or forcing soil against a hard surface such as a sample trowel to ensure that headspace is eliminated.

7. Use a paper towel to remove soil from the side of the sampler so a tight seal can be made between the sample cap and the rubber O-ring.

8. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler.

9. Remove any surface soil from the outside of the sampler and place in the foil bag provided with the sampler. Good work hygiene practices and diligent decontamination procedures prevents the spread of contamination even on the outside of the containers.

10. Label the bag with appropriate information in accordance with SOP SA-6.3.

11. Place the full sampler inside a lined cooler with ice and cool to 4 °C ± 2 °C. Make sure any required trip blanks and temperature blanks are also in the cooler. Secure custody of the cooler in accordance with SOP SA-6.3.

12. Typically, collect three Encore™ samplers at each location. Consult the SAP or laboratory to determine the required number of Encore™ samplers to be collected.

13. The T-handle shall be decontaminated before moving to the next interval or location using a soap and water wash and rinse, and where applicable, the selected solvent as defined in the project planning documents.

Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler.

After the Encore™samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

6.2.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analyses using both the low-level (sodium bisulfate preservation) and high- to medium-level (methanol preservation) methods.

Safety Reminder

When using chemicals in the field to preserve samples, the FOL and/or SSO must ensure that Materials Safety Data Sheets (MSDSs) have been provided with the chemicals to be used. They also must ensure that these chemicals have been added to the Chemical Inventory List contained within Section 5.0, Hazard Communication, of your Health and Safety Guidance Manual (HSGM). Lastly, but most importantly, the FOL and/or SSO must review the hazards with personnel using these chemicals and ensure that provisions are available for recommended PPE and emergency measures (e.g., eyewash, etc.).

Methanol Preservation (High to Medium Level):

Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40 to 60 mL glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

CAUTION

NEVER attempt to pipette by mouth

In situations where personnel are required to spike the septum using a hypodermic needle, the following provisions for handling sharps must be in place:

- Training of personnel regarding methods for handling of sharps
- Hard-sided containers for the disposal of sharps
- Provisions for treatment in cases where persons have received a puncture wound

Soil shall be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable tube/plunger-type syringe with the tip cut off. The outside diameter of the coring device must be smaller than the inside diameter of the sample bottle neck.

A small electronic balance or manual scale will be necessary for measuring the volume of soil to be added to the methanol-preserved sample bottle. Calibration of the scale shall be performed prior to use and intermittently throughout the day according to the manufacturer's requirements.

The sample should be collected as follows:

1. Weigh the unused syringe and plunger to the nearest 0.01 gram.
2. Pull the plunger back and insert the syringe into the soil to be sampled.
3. Collect 8 to 12 grams of soil by pushing the syringe barrel into the soil.
4. Weigh the sample and adjust until obtaining the required amount of sample.

5. Record the sample weight to the nearest 0.01 gram in the field logbook and/or on the sample log sheet.

6. Extrude the weighed soil sample into the methanol-preserved sample bottle taking care not to contact the sample container with the syringe.

7. If dirty, wipe soil particles from the threads of the bottle and cap. Cap the bottle tightly.

8. After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol.

9. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

Sodium Bisulfate Preservation (Low Level):

> ***CAUTION***
> Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soil containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode. To avoid this hazard or hazards of this type, a small sample aliquot should be subjected to the sample preservative. If it effervesces in an open air environment, utilize an alternative method such as Encore™ or 2-ounce jar.

Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be preserved in the field using the sodium bisulfate method are to be prepared and collected as follows:

1. Add 1 gram of sodium bisulfate to 5 mL of laboratory-grade deionized water in a 40 to 60 mL glass vial with septum-lined lid.

2. Collect the soil sample and record the sample weight to the nearest 0.01 gram in the field logbook or on the sample log sheet as described for methanol preservation

3. Add the weighed sample to the sample vial.

4. Collect duplicate samples using the methanol preservation method on a one-for-one sample basis because it is necessary for the laboratory to perform both low-level and medium-level analyses.

5. Place the samples on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

> ***NOTE***
> If lower detection limits are necessary, an option to field preserving with sodium bisulfate may be to collect EnCore™ samplers at a given sample location. Consult the planning documents to determine whether this is required. If it is, collect samples in accordance with the Encore™ sampling procedure above and then send all samplers to the laboratory to perform the required preservation and analyses.

6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses

Samples collected for non-volatile analyses may be collected as either grab or composite samples as follows:

1. With a stainless steel trowel or other approved tool, transfer a portion of soil to be sampled to a stainless steel bowl or disposable inert plastic tray.

2. Remove roots, vegetation, sticks, and stones larger than the size of a green pea.

3. Thoroughly mix the soil in the bowl or tray to obtain as uniform a texture and color as practicable. The soil type, moisture content, amount of vegetation, and other factors may affect the amount of time required to obtain a properly mixed sample. In some cases, it may be impossible to obtain a uniform sample appearance. Use the field logbook to describe any significant difficulties encountered in obtaining a uniform mixture.

4. Transfer the mixed soil to the appropriate sample containers and close the containers.

5. Label the sample containers in accordance with SOP SA-6.3.

6. Place the containers in a cooler of ice as soon after collection as possible.

7. Prepare the sample shipment and ship the samples in accordance with SOP SA-6.1.

> ***NOTE***
> Cooling may not be required for some samples depending on the scheduled analyses. Consult the planning documents if in doubt regarding correct sample preservation conditions. When in doubt – Cool to 4°C.

> ***NOTE***
> Head space is permitted in soil sample containers for non-volatile analyses to allow for sample expansion.

6.2.3 Procedure for Collecting Undisturbed Soil Samples

> ***NOTE***
> Use of thin-walled undisturbed tube samplers is restricted by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soil with a consistency in excess of very stiff cannot be penetrated by the sampler. Devices such as Dennison or Pitcher core samplers can be used to obtain undisturbed samples of stiff soil. Using these devices normally increases sampling costs, and therefore their use should be weighed against the need for acquiring an undisturbed sample. These devices are not discussed in this SOP because they are not commonly used.

When it is necessary to acquire undisturbed samples of soil for purposes of engineering parameter analysis (e.g., permeability), a thin-walled, seamless tube sampler (Shelby tube) shall be employed using the following collection procedure:

1. In preparation for sampling utilizing a drill rig, field personnel must complete the following activities:

   - Ensure that all subsurface drilling activities are preceded by a utility clearance for the area to be investigated. This includes activities described in SOP HS-1.0, Utility Location and Excavation Clearance, as well as any location-specific procedures that may apply.

REMEMBER

If you are digging near a marked utility (within the diameter of an underground utility that has been marked plus 18 inches), you must first locate the utility through vacuum extraction or hand digging to ensure that your activities will not damage the utility.

- Complete an Equipment Inspection Checklist for the drill rig or direct-push technology (DPT) rig. This checklist will be provided in the HASP.
- Review the Safe Work Permit prior to conducting the activity.
- Review the activity to be conducted.

2. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and/or clean out the borehole to the desired sampling depth. Be careful to minimize potential disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

CAUTION

The use of bottom-discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Only the use of side-discharge bits is permitted.

3. Determine whether a stationary piston-type sampler is required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used.

4. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal. In addition, the check valve maintains a positive suction within the tube to help retain the sample.

5. A stainless steel tube sampler is typically used to minimize chemical reaction between the sample and the sampling tube.

6. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil with a continuous and rapid motion, without impacting or twisting. If the soil is too hard to penetrate by pushing alone, careful hammering may be used by minimizing drop distance (tapping) of the hammer. Before pulling the tube, turn it at least one revolution to shear the sample off at the bottom. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.

7. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated.

8. Remove disturbed material in the upper end of the tube and measure the length of sample again.

9. After removing at least 1 inch of soil from the lower end, place enough packing material (clean inert material such as paper or cloth) tightly in each end of the Shelby tube and then pour melted wax into each end to make at least a ½-inch wax plug and then add more packing material to fill the voids at both ends.

10. Place plastic caps on the ends, tape the caps in place, and dip the ends in wax to prevent loss of soil.

11. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label.

12. Mark the "up" direction on the side and upper end of the tube with indelible ink.

13. Complete a chain-of-custody form (see SOP SA-6.3) and other required forms (including Attachment A of this SOP).

14. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

CAUTION

To preserve sample integrity do not allow tubes to freeze, and store the samples vertically with the same orientation they had in the ground, (i.e., top of sample is up) in a cool place out of the sun at all times.

CAUTION

A primary concern in the preparation of the wax plugs is the potential for the heat source and melted wax to cause a fire and/or burns. Follow the directions below to prevent injury or fire.

Electrical Heating

Using hot plates to melt the wax is acceptable. In an outdoor setting, make sure a Ground Fault Circuit Interrupter (GFCI) is employed within the electrical circuit. If a portable generator is used, ensure that the generator is an adequate distance from the sampling operation (at least 50 feet). Ensure that the extension cord is rated for the intended load and for outdoor use and is free from recognizable damage. Ensure flammable preservatives are not employed or stored near the hot plate. Although a Hot Work Permit is not required, scene safety evaluation by site personnel of the above elements is. As always, if a fire potential exists, the provisions for extinguishing must be immediately accessible as well as any provisions for first aid measures.

Open Flame

If an open flame is used, the following provisions are necessary:

- Complete a Hot Work Permit and any local permit required for elevated temperature applications. The Hot Work Permit, provided in your HASP, will aid the FOL and/or the SSO in ensuring that fire protection provisions (extinguishers, fire watches, etc.) are in place as well as ensuring that local requirements have been addressed.

- Ensure that water is available to address any wax splashes or contact. If possible, immerse the contacted area. Where this is not possible, run water over the area and apply cold compresses. The need for medical attention or first aid shall be determined on site under the direction of the SSO.

6.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples for subsequent analysis is by use of a stainless steel shovel, hand auger, soil corer, or stainless steel or disposable plastic trowel.

> ***NOTE***
> Multiple depth intervals are used to describe surface soil. Sometimes surface soil is defined as soil from 0 to 2 inches below ground surface (bgs), and sometimes it is defined as soil from other depths such as 0 to 2 feet bgs. Ensure that the definition of surface soil depth is clear before collecting surface soil samples.
>
> For the purposes of instruction, the terms "surface soil" and "near-surface soil" are used in this SOP as follows:
>
> - Surface soil - 0 to 6 inches bgs
> - Near-surface soil - 6 to 18 inches bgs
>
> If these intervals are defined differently in the planning documents, substitute the appropriate depth ranges.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Stainless steel hand auger, soil corer, or shovel.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in project planning document.
- Required PPE.
  - Nitrile surgeon's or latex gloves may be used, layered as necessary.
  - Safety glasses
  - Other – Items identified on the Safe Work Permit may be required based on location-specific requirements such as hearing protection, steel-toed work boots, and a hard hat when working near a drill rig. These provisions will be listed in the HASP or directed by the FOL and/or SSO.

> ***Safety Reminder***
> The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP)
- Required decontamination equipment
- Required sample container(s)
- Wooden stakes or pin flags

- Sealable polyethylene bags (e.g., Ziploc® baggies)
- Heavy duty cooler
- Ice
- Chain-of-custody records and custody seals

When acquiring surface soil samples, use the following procedure:

1. Place padding or use knee pads when kneeling near the sample location. If necessary, place plastic sheeting to provide a clean surface for sample equipment to avoid possible cross- contamination.
2. Carefully remove vegetation, roots, twigs, litter, etc. to expose an adequate soil surface area to accommodate sample volume requirements.
3. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting surface soil samples for volatile analysis. Surface soil samples for volatile organic analysis should be collected deeper than 6 inches bgs because shallower material has usually lost most of the volatiles through evaporation. Ensure that the appropriate surface soil depth is being analyzed in accordance with the planning document.
4. Using decontaminated sampling tools, thoroughly mix in place a sufficient amount of soil to fill the remaining sample containers. See Section 6.5 of this procedure for hand auger instruction, as needed.
5. Transfer the sample into those containers utilizing a stainless steel trowel.
6. Cap and securely tighten all sample containers.
7. Affix a sample label to each container. Be sure to fill out each label carefully and clearly, addressing all the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.
9. Site restoration – Whenever removing sample materials, always restore the surface. It is our intent to leave the area better than we found it. Do NOT create trip hazards in areas when pedestrian traffic may exist.

6.4 Near-Surface Soil Sampling

Collection of samples from near the surface (depth of 6 to 18 inches) can be accomplished with tools such as shovels, hand auger, soil corers, and stainless steel or pre-cleaned disposable trowels and the equipment listed under Section 6.5 of this procedure.

To obtain near-surface soil samples, the following protocol shall be used:

1. With a clean shovel, make a series of vertical cuts in the soil to the depth required to form a square approximately 1 foot by 1 foot.
2. Lever out the formed plug and scrape the bottom of the freshly dug hole with a decontaminated stainless steel or pre-cleaned disposable trowel to remove any loose soil.

3. Follow steps 1 through 9 of Section 6.3.

6.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of stainless steel bucket bits (approximately 6.5 inches long and 2, 2.75, 3.25, and 4 inches in diameter), series of extension rods (available in 2-, 3-, 4- and 5-inch lengths), and a T-handle connected to extension rods and to the auger bucket. A larger-diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then it is withdrawn. The larger-diameter bit is then replaced with a smaller-diameter bit, lowered down the hole, and slowly turned into the soil to the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil either from the surface, or to depths in excess of 12 feet. However, the presence of subsurface rocks and landfill material and collapse of the borehole normally limit sampling depth.

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes)
- Stainless steel mixing bowls
- The equipment listed in Section 6.3
- Miscellaneous hand tools as required to assemble and disassemble the hand auger units

CAUTION

Potential hazards associated with hand augering include:

- Muscle strain and sprain due to over twisting and/or over compromising yourself.
- Equipment failure due to excessive stress on the T-handle or rods through twisting. Failure of any of these components will result in a sudden release and potential injury due to that failure.

As in all situations, any intrusive activities that could damage underground utilities shall be proceeded by a Dig/Excavation permit/ticket. Call the Utility Locating service in the area or your Project Health and Safety Officer for more information. When in doubt – **Get the Ticket!**

To obtain soil samples using a hand auger, use the following procedure:

1. Wearing designated PPE, attach a properly decontaminated bucket bit to a clean extension rod and attach the T-handle to the extension rod.
2. Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.).
3. Twist the bucket into the ground while pushing vertically downward on the auger. The cutting shoes fill the bucket as it is advanced into the ground.
4. As the auger bucket fills with soil, periodically remove any unneeded soil.

5. Add rod extensions as necessary to extend the reach of the auger. Also, note (in a field notebook, boring log, and/or on a standardized data sheet) any changes in the color, texture or odor of the soil as a function of depth. The project-specific planning document (SAP, HASP, etc.) describe requirements for scanning the soil with a real-time air monitoring instrument (e.g., PID, FID, etc.) and recording the measurements.

6. After reaching the desired depth (e.g., the top of the interval to be sampled), slowly and carefully withdraw the apparatus from the borehole to prevent or minimize movement of soil from shallower intervals to the bottom of the hole.

7. Remove the soiled bucket bit from the rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is to be smaller in diameter than the bucket bit employed to initiate the borehole.

8. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.

9. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.

10. Discard the top of the core (approximately 1 inch), which represents any loose material collected by the bucket bit before penetrating the sample material.

11. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting a soil sample for volatile compound analysis directly from the bucket bit.
12. Utilizing a properly decontaminated stainless steel trowel or dedicated disposable trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl.

13. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers. Refer to Section 6.2.2.

14. Follow steps 4 through 7 listed in Section 6.3.

6.5.1 Sampling Using Stainless Steel Soil Corers

A soil corer is a stainless steel tube equipped with a cutting shoe and sample window in the side. The soil corer is advanced into the soil by applying downward pressure (body weight). The soil is unloaded by then forcing a ram towards the cutting shoe, which results in the discharge of the soil core through a window in the sleeve.

Use, application, and sample protocol is the same as for hand augering provided above, but without necessarily rotating the corer while advancing it.

SAFETY REMINDER

Hand augering and soil corer sampling can be physically demanding based on the type of geology and subsurface encumbrances encountered. Soil coring has some added hazards such the corer collapsing under your weight. To reduce the potential for muscle strain and damage, the following measures will be incorporated:

- Stretch and limber your muscles before heavy exertion. This hazard becomes more predominant in the early morning hours (prior to muscles becoming limber) and later in the day (as a result of fatigue).

- Job rotation – Share the duties so that repetitive actions do not result in fatigue and injury.
- Increase break frequencies as needed, especially as ambient conditions of heat and/or cold stress may dictate.
- Do not force the hand tools or use cheater pipes or similar devices to bypass an obstruction. Move to another location near the sampling point. Exerting additional forces on the sampling devices can result in damage and/or failure that could potentially injure someone in the immediate vicinity.
- Do not over compromise yourself when applying force to the soil corer or hand auger. If there is a sudden release, it could result in a fall or muscle injury due to strain.

6.6 Subsurface Soil Sampling with a Split-Barrel Sampler

A split-barrel (split-spoon) sampler consists of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-pound or larger casing driver.

Safety Reminder

It is intended through the Equipment Inspection for Drill Rigs form provided in the HASP that the hammer and hemp rope, where applicable, associated with this activity will be inspected (no physical damage is obvious), properly attached to the hammer (suitable knots or sufficient mechanical devices), and is in overall good condition.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (2-inch OD, 1-3/8-inch ID, either 20 inches or 26 inches long); Larger OD samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-pound weight, driving head, and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.
- Equipment listed in Section 6.3.

The following steps shall be followed to obtain split-barrel samples (Steps 1 through 4 are typically performed by the drilling subcontractor):

1. Attach the split-barrel sampler to the sampling rods.

2. Lower the sampler into the borehole inside the hollow stem auger bits.

3. Advance the split-barrel sampler by hammering the length (typically 18 or 24 inches) of the split-barrel sampler into the soil using 140-pound or larger hammer.

4. When the desired depth is achieved, extract the drill rods and sampler from the augers and/or borehole.

5. Detach the sampler from the drill rods.

6. Place the sampler securely in a vise so it can be opened using pipe wrenches.

> ***CAUTION***
> Pipe wrenches are used to separate the split spoon into several components. The driller's helper should not apply excessive force through the use of cheater pipes or push or pull in the direction where, if the wrench slips, hands or fingers will be trapped against an immovable object.

7. Remove the drive head and nosepiece with the wrenches, and open the sampler to reveal the soil sample.

8. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.) (as project-specific planning documents dictate). Carefully separate (or cut) the soil core, with a decontaminated stainless steel knife or trowel, at about 6-inch intervals while scanning the center of the core for elevated readings. Also scan stained soil, soil lenses, and anomalies (if present), and record readings.

9. If elevated vapor readings were observed, collect the sample scheduled for volatile analysis from the center of the core where elevated readings occurred. If no elevated readings where encountered, the sample material should be collected from the core's center (this area represents the least disturbed area with minimal atmospheric contact) (refer to Section 6.2.1).

10. Using the same trowel, remove remaining sample material from the split-barrel sampler (except for the small portion of disturbed soil usually found at the top of the core sample) and place the soil into a decontaminated stainless steel mixing bowl.

11. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers (refer to Section 6.2.2).

12. Follow steps 4 through 7 in Section 6.3.

6.7 Subsurface Soil Sampling Using Direct-Push Technology

Subsurface soil samples can be collected to depths of 40+ feet using DPT. DPT equipment, responsibilities, and procedures are described in SOP SA-2.5.

6.8 Excavation and Sampling of Test Pits and Trenches

6.8.1 Applicability

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

CAUTION

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise from the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden, steel, or aluminum support structures or through sloping and benching. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments; therefore, monitoring will be conducted by the Competent Person to determine if it is safe to enter. Any entry into a trench greater than 4 feet deep will constitute a Confined Space Entry and must be conducted in conformance with OSHA standard 29 CFR 1910.146. In all cases involving entry, substantial air monitoring, before entry, appropriate respiratory gear and protective clothing determination, and rescue provisions are mandatory. There must be at least three people present at the immediate site before entry by one of the field team members. This minimum number of people will increase based on the potential hazards or complexity of the work to be performed. The reader shall refer to OSHA regulations 29 CFR 1926.650, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146. High-hazard entries such as this will be supported by members of the Health Sciences Group professionally trained in these activities.

Excavations are generally not practical where a depth of more than about 15 to 20-feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If soil data at depths greater than 15-feet are required, the data are usually obtained through test borings instead of test pits.

In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations.

6.8.2 Test Pit and Trench Excavation

Test pits or trench excavations are constructed with the intent that they will provide an open view of subsurface lithology and/or disposal conditions that a boring will not provide. These procedures describe the methods for excavating and logging test pits and trenches installed to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).

Test pits and trenches may be excavated by hand or power equipment to permit detailed descriptions of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:

- The purpose and extent of the exploration

- The space required for efficient excavation
- The chemicals of concern
- The economics and efficiency of available equipment

Test pits normally have a cross section that is 4 to 10 feet square; test trenches are usually 3 to 6 feet wide and may be extended for any length required to reveal conditions along a specific line. The following table provides guidelines for design consideration based on equipment efficiencies.

Equipment	Typical Widths, in Feet
Trenching machine	0.25 to 1.0
Backhoe/Track Hoe	2 to 6

The lateral limits of excavation of trenches and the position of test pits shall be carefully marked on area base maps. If precise positioning is required to indicate the location of highly hazardous materials, nearby utilities, or dangerous conditions, the limits of the excavation shall be surveyed. Also, if precise determination of the depth of buried materials is needed for design or environmental assessment purposes, the elevation of the ground surface at the test pit or trench location shall also be determined by survey. If the test pit/trench will not be surveyed immediately, it shall be backfilled and its position identified with stakes placed in the ground at the margin of the excavation for later surveying.

The construction of test pits and trenches shall be planned and designed in advance as much as possible. However, the following field conditions may necessitate revisions to the initial plans:

- Subsurface utilities
- Surface and subsurface encumbrances
- Vehicle and pedestrian traffic patterns
- Purpose for excavation (e.g., the excavation of potential ordnance items)

The final depth and construction method shall be collectively determined by the FOL and designated Competent Person. The actual layout of each test pit, temporary staging area, and spoils pile may further be predicated based on site conditions and wind direction at the time the test pit is excavated. Prior to excavation, the area may be surveyed by magnetometer or metal detector or other passive methods specified in SOP HS1.0, Utility Location and Excavation Clearance, to identify the presence of underground utilities or drums. Where possible, the excavator should be positioned upwind and preferably within an enclosed cab.

No personnel shall enter any test pit or excavation except as a last resort, and then only under direct supervision of a Competent Person. If entrance is required, OSHA requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be placed for every 25 feet of lateral travel and extended 3 feet above ground surface). A temporary guard rail or vehicle stop must be placed along the surface of the hole before entry in situations where the excavation may be approached by traffic. Spoils will be stockpiled no closer than 2 feet from the sidewall of the excavation. The excavation equipment operator shall be careful not to undercut sidewalls and will, where necessary, bench back to increase stability. The top cover, when considered clean, will be placed separately from the subsurface materials to permit clean cover. It is emphasized that the project data needs should be structured such that required samples can be collected without requiring entrance into the excavation. For example,

samples of leachate, groundwater, or sidewall soil can be collected with telescoping poles or similar equipment.

Dewatering and watering may be required to ensure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation stable. This is an important consideration for excavations in cohesionless material below the groundwater table and for excavations left open greater than a day. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.

Where possible excavations and test pits shall be opened and closed within the same working day. Where this is not possible, the following engineering controls shall be put in place to control access:

- Trench covers/street plates
- Fences encompassing the entire excavation intended to control access
- Warning signs warning personnel of the hazards
- Amber flashing lights to demarcate boundaries of the excavation at night

Excavations left open will have emergency means to exit should someone accidentally enter.

6.8.3 Sampling in Test Pits and Trenches

6.8.3.1 General

Log test pits and trenches as they are excavated in accordance with the Test Pit Log presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.

Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable health and safety and OSHA requirements have been met as stated above. These provisions will be reiterated as appropriate in the project-specific HASP.

The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.

In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information includes soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples that can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost-effective method of sampling than installing borings.

6.8.3.2 Sampling Equipment

The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:

- Backhoe or other excavating machinery.
- Shovels, picks, hand augers, and stainless steel trowels/disposable trowels.
- Sample container - bucket with locking lid for large samples; appropriate bottle ware for chemical or geotechnical analysis samples.
- Polyethylene bags for enclosing sample containers; buckets.
- Remote sampler consisting of 10-foot sections of steel conduit (1-inch-diameter), hose clamps, and right angle adapter for conduit (see Attachment D).

6.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 6.8.3.4.

- Excavate the trench or pit in several 0.5- to 1.0-foot depth increments. Where soil types support the use of a sand bar cutting plate, use of this device is recommended to avoid potentially snagging utilities with the excavator teeth. It is recommended that soil probes or similar devices be employed where buried items or utilities may be encountered. This permits the trench floor to be probed prior to the next cut.
- After each increment:
  - the operator shall wait while the sampler inspects the test pit from grade level
  - the sampler shall probe the next interval where this is considered necessary. Practical depth increments for lithological evaluations may range from 2 to 4 feet i or where lithological changes are noted.
- The backhoe operator, who will have the best view of the test pit, shall immediately cease digging if:
  - Any fluid phase, including groundwater seepage, is encountered in the test pit
  - Any drums, other potential waste containers, obstructions, or utility lines are encountered
  - Distinct changes of material being excavated are encountered

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending on the conditions encountered, it may be required to excavate more slowly and carefully with the backhoe.

For obtaining test pit samples from grade level, the following procedure shall be followed:

- Use the backhoe to remove loose material from the excavation walls and floor to the greatest extent possible.
- Secure the walls of the pit, if necessary. (There is seldom any need to enter a pit or trench that would justify the expense of shoring the walls. All observations and samples should be taken from the ground surface.)

- Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material after it has been deposited on the ground, as follows:

  a. The sampler or FOL shall direct the backhoe operator to remove material from the selected depth or location within the test pit/trench.

  b. The backhoe operator shall bring the bucket over to a designated location on the sidewall a sufficient distance from the pit (at least 5 feet) to allow the sampler to work around the bucket.

  c. After the bucket has been set on the ground, the backhoe operator shall either disengage the controls or shut the machine down.

  d. When signaled by the operator that it is safe to do, the sampler will approach the bucket.

  e. The soil shall be monitored with a photoionization or flame ionization detector (PID or FID) as directed in the project -specific planning documents.

  f. The sampler shall collect the sample from the center of the bucket or pile in accordance with surface soil sampling procedures of Section 6.3 or 6.4, as applicable. Collecting samples from the center of a pile or bucket eliminates cross-contamination from the bucket or other depth intervals.

- If a composite sample is desired, several depths or locations within the pit/trench will be selected, and the bucket will be filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.

> ***CAUTION***
>
> Care must be exercised when using the remote sampler described in the next step because of potential instability of trench walls. In situations where someone must move closer than 2 feet to the excavation edge, a board or platform should be used to displace the sampler's weight to minimize the chance of collapse of the excavation edge. Fall protection should also be employed when working near the edges or trenches greater than 6 feet deep. An immediate means to extract people who have fallen into the trench will be immediately available. These means may include ladders or rope anchor points.

- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the sidewall or bottom of the pit as follows:

  a. Scrape the face of the pit/trench using a long-handled shovel or hoe to remove the smeared zone that has contacted the backhoe bucket.

  b. Collect the sample directly into the sample jar, by scraping with the jar edge, eliminating the need for sample handling equipment and minimizing the likelihood of cross-contamination.

  c. Cap the sample jar, remove it from the remote sampler assembly, and package the sample for shipment in accordance with SOP SA-6.3.

- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

6.8.3.4 In-Pit Sampling

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of soil or wastes within the test pit/trench) or when samples from relatively small discrete zones within the test pit are required. This approach may also be necessary to sample any seepage occurring at discrete levels or zones in the test pit that are not accessible with remote samplers.

In general, personnel shall sample and log pits and trenches from the ground surface, except as provided for by the following criteria:

- There are no practical alternative means of obtaining such data.
- The SSO and Competent Person determine that such action can be accomplished without breaching site safety protocol. This determination will be based on actual monitoring of the pit/trench after it is dug (including, at a minimum, measurements of oxygen concentration, flammable gases, and toxic compounds, in that order). Action levels will be provided in project-specific planning documents.
- A company-designated Competent Person determines that the pit/trench is stable trough soil classification evaluation/inspections or is made stable (by cutting/grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements shall be strictly observed.

If these conditions are satisfied, only one person may enter the pit/trench. On potentially hazardous waste sites, this individual shall be dressed in selected PPE as required by the conditions in the pit. He/she shall be affixed to a harness and lifeline and continuously monitored while in the pit.

A second and possible third individual shall be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations to support self rescue or assisted self rescue. The individual entering the pit shall remain therein for as brief a period as practical, commensurate with performance of his/her work. After removing the smeared zone, samples shall be obtained with a decontaminated trowel or spoon.

6.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 6.8.3.2, the following equipment is needed for geotechnical sampling:

- Soil sampling equipment, similar to that used in shallow drilled boring (i.e., thin-walled tube samplers), that can be pushed or driven into the floor of the test pit.
- Suitable driving (e.g., sledge hammer) or pushing (e.g., backhoe bucket) equipment used to advance the sampler into the soil.
- Knives, spatulas, and other suitable devices for trimming hand-carved samples.
- Suitable containers (bags, jars, tubes, boxes, etc.), labels, wax, etc. for holding and safely transporting collected soil samples.
- Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

Disturbed grab or bulk geotechnical soil samples may be collected for most soil in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic-lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification: larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soil using thin-walled tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability, and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe, rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the tube when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 6.8.3.4 shall be followed. The thin-walled tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate because the sample will not have the correct orientation.

A sledge hammer or backhoe may be used to drive or push the tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. When using a sledge hammer, it is recommended that the sampler be stabilized using a rope/strap wrench or pipe wrench to remove the person's hands holding the sampler from the strike zone. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hook the sampler to the excavator or backhoe and extract. This means an alternative head will be used as a connection point or that multiple choke hitches will be applied to extract the sampler. If this fails and the excavator can dig deeper without potentially impacting subsurface utilities, excavate the sampler. If this fails or if the excavator cannot be used due to subsurface utilities, hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry into the test pit, the requirements in Section 6.8.3.4 must be followed. Prepare the sample as described in Steps 9 through 13 in Section 6.2.3, and label, pack and transport the sample in the required manner, as described in SOPs SA-6.3 and SA-6.1.

6.8.4 Backfilling of Trenches and Test Pits

All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.

Before backfilling, the onsite crew may photograph, if required by the project-specific work plan, all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.

After inspection, backfill material shall be returned to the pit under the direction of the FOL. Backfill should be returned to the trench or test pit in 6-inch to 1-foot lifts and compacted with the bucket. Remote controlled tampers or rollers may be lowered into the trench and operated from top side. This procedure will continue to the grade surface. It is recommended that the trench be tracked or rolled in. During excavation, clean soil from the top 2 feet may have been separated to be used to cover the last segments. Where these materials are not clean, it is recommended that clean fill be used for the top cover.

If a low-permeability layer is penetrated (resulting in groundwater flow from an upper contaminated flow zone into a lower uncontaminated flow zone), backfill material must represent original conditions or be impermeable. Backfill could consist of a soil-bentonite mix prepared in a proportion specified by the FOL (representing a permeability equal to or less than original conditions). Backfill can be covered by "clean" soil and graded to the original land contour. Revegetation of the disturbed area may also be required.

6.9 Records

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler for all samples collected. All soil sampling locations should be documented by tying in the location of two or more nearby permanent landmarks (building, telephone pole, fence, etc.) or obtaining GPS coordinates; and shall be noted on the appropriate sample log sheet, site map, or field notebook. Surveying may also be necessary, depending on the project requirements.

Test pit logs (see Attachment C of this SOP) shall contain a sketch of pit conditions. If the project-specific work plan requires photographs, at least one photograph with a scale for comparison shall be taken of each pit. Included in the photograph shall be a card showing the test pit number. Boreholes, test pits, and trenches shall be logged by the field geologist in accordance with SOP GH-1.5.

Other data to be recorded in the field logbook include the following:

- Name and location of job
- Date of boring and excavation
- Approximate surface elevation
- Total depth of boring and excavation
- Dimensions of pit
- Method of sample acquisition
- Type and size of samples
- Soil and rock descriptions
- Photographs if required
- Groundwater levels
- PID/FID/LEL/O_2 meter readings
- Other pertinent information, such as waste material encountered

In addition, site-specific documentation to be maintained by the SSO and/or Competent Person will be required including:

- Calibration logs
- Excavation inspection checklists

- Soil type classification

7.0 REFERENCES

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ATTACHMENT A
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

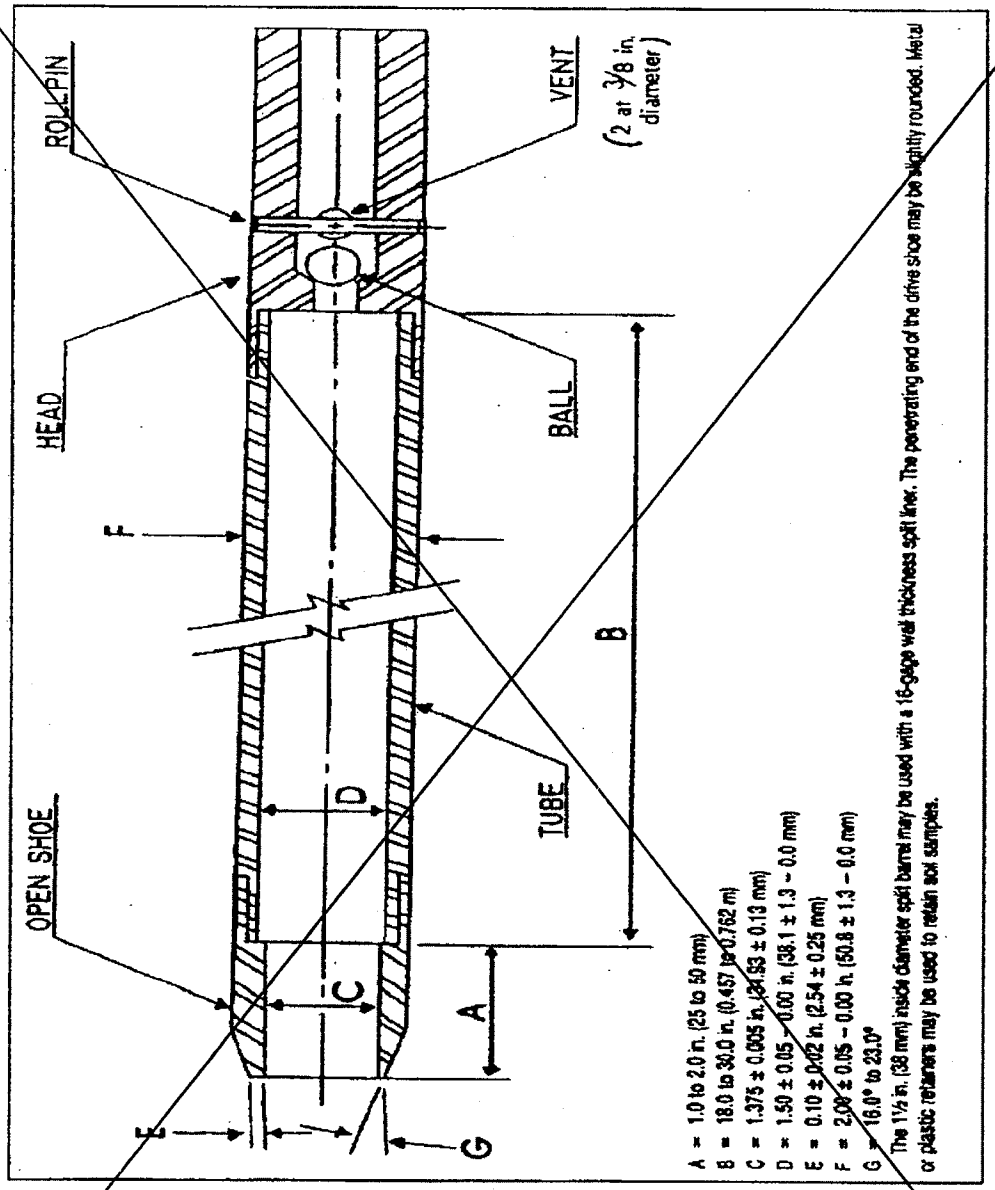
MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SPLIT-SPOON SAMPLER



A = 1.0 to 2.0 in. (25 to 50 mm)
B = 18.0 to 30.0 in. (0.457 to 0.762 m)
C = 1.375 ± 0.005 in. (34.93 ± 0.13 mm)
D = 1.50 ± 0.05 – 0.00 in. (38.1 ± 1.3 – 0.0 mm)
E = 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
F = 2.00 ± 0.05 – 0.00 in. (50.8 ± 1.3 – 0.0 mm)
G = 16.0° to 23.0°

The 1½ in. (38 mm) inside diameter split barrel may be used with a 16-gage wall thickness split liner. The penetrating end of the drive shoe may be slightly rounded. Metal or plastic retainers may be used to retain soil samples.

ATTACHMENT C
TEST PIT LOG

Tetra Tech NUS, Inc.

TEST PIT LOG

Page ___ of ___

PROJECT NAME: _____ TEST PIT No.: _____
PROJECT NUMBER: _____ DATE: _____
LOCATION: _____ GEOLOGIST: _____

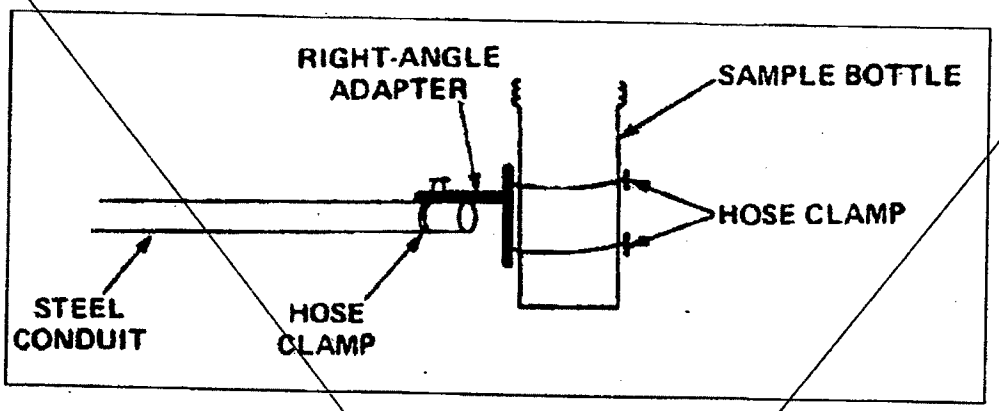
Depth (Ft.)	Lithology Change (Depth/Ft.)	MATERIAL DESCRIPTION Soil/Waste Characteristics (lithology, density, color, etc.)	USCS	Remarks	PID/FID READING Source (ppm)	BZ (ppm)

TEST PIT CROSS SECTION AND / OR PLAN VIEW

REMARKS: _____

PHOTO LOG: _____

ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING



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1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

5.0 SOIL SAMPLING PROCEDURES

5.1 General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

5.2 Sampling Equipment

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

5.3 DPT Sampling Methodology

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.

- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.

- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.

- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.

- Place the acetate liner containing the soils in the trough.

- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.

- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.

- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).

- Sampling equipment is decontaminated prior to collecting the next sample.

~~6.0 GROUNDWATER SAMPLING PROCEDURES~~

6.1 General

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.

Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

6.2 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:

- 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point
- Connecting rods
- Roto-hammer with 1.5-inch bit
- Mechanical jack
- 1/4-inch OD polyethylene tubing
- 3/8-inch OD polyethylene tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.

- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.

- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity (±10 percent), the well may be sampled.

- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.

- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).

- Decontaminate the equipment before moving to the next location.

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

ATTACHMENT 1
SAFE WORK PERMIT FOR DPT OPERATIONS

Permit No. _____ Date: _____ Time: From _____ to _____

SECTION I: General Job Scope

I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**

II. Required Monitoring Instruments: _____

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____ _____ TtNUS

SECTION II: General Safety Requirements (To be filled in by permit issuer)

V. Protective equipment required

Level D ☒ Level B ☐
Level C ☐ Level A ☐
Detailed on Reverse

Respiratory equipment required

Full face APR ☐ Escape Pack ☐
Half face APR ☐ SCBA ☐
SKA-PAC SAR ☐ Bottle Trailer ☐
Skid Rig ☐ None ☒

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

VI. Chemicals of Concern _____ Action Level(s) _____ Response Measures _____

VII. Additional Safety Equipment/Procedures

Hard-hat	☒ Yes	☐ No	Hearing Protection (Plugs/Muffs)	☒ Yes	☐ No
Safety Glasses	☒ Yes	☐ No	Safety belt/harness	☐ Yes	☒ No
Chemical/splash goggles	☐ Yes	☒ No	Radio	☐ Yes	☒ No
Splash Shield	☐ Yes	☒ No	Barricades	☒ Yes	☐ No
Splash suits/coveralls	☐ Yes	☐ No	Gloves (Type - _____)	☐ Yes	☐ No
Steel toe Work shoes or boots	☒ Yes	☐ No	Work/warming regimen	☐ Yes	☐ No

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors

	Yes	NA		Yes	NA
Safety shower/eyewash (Location & Use)	☐	☒	Emergency alarms	☐	☐
Daily tail gate meetings	☒	☐	Evacuation routes	☐	☐
Contractor tools/equipment/PPE inspected	☐	☐	Assembly points	☐	☐

IX. Site Preparation

Utility Clearances obtained for areas of subsurface investigation	☐ Yes	☐ No
Physical hazards removed or blockaded	☐ Yes	☐ No
Site control boundaries demarcated/signage	☐ Yes	☐ No

X. Equipment Preparation

	Yes	NA
Equipment drained/depressurized	☐	☒
Equipment purged/cleaned	☐	☒
Isolation checklist completed	☐	☒
Electrical lockout required/field switch tested	☐	☒
Blinds/misalignments/blocks & bleeds in place	☐	☒
Hazardous materials on walls/behind liners considered	☐	☒

XI. Additional Permits required (Hot work, confined space entry). ☐ Yes ☐ No

If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions: _____

Permit Issued by: _____ Permit Accepted by: _____

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (~~y~~261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (~~y~~261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration			Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER						
Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days
SOIL						
Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR						
Volatile Organics	Low/Medium		Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2 [9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5 [10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11],[12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11],[14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs and reports generally initiated and maintained for documenting Tetra Tech NUS field activities.

2.0 SCOPE

Documents presented within this procedure (or equivalents) shall be used for all Tetra Tech NUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

Project Manager (PM) - The Project Manager is responsible for obtaining hardbound, controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The Field Operations Leader is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports illustrated in this guideline (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time-frame.

5.0 PROCEDURES

5.1 Site Logbook

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major onsite activities are documented. At a minimum, the following activities/events shall be recorded or referenced (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Time and date of H&S training
- Arrival/departure of equipment
- Time and date of equipment calibration
- Start and/or completion of borehole, trench, monitoring well installation, etc.
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day

that onsite activities take place which involve Tetra Tech NUS or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project's central file.

The following information must be recorded on the cover of each site logbook:

- Project name
- Tetra Tech NUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2), but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the field notebook in which the measurements are recorded (see Attachment A).

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

When movies, slides, or photographs are taken of a site or any monitoring location, they must be numbered sequentially to correspond to logbook/notebook entries. The name of the photographer, date, time, site location, site description, and weather conditions must be entered in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided, since they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend upon the subject matter, type of camera (digital or film), and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Once processed, the slides of photographic prints shall be consecutively numbered and labeled according to the logbook/notebook descriptions. The site photographs and associated negatives and/or digitally saved images to compact disks must be docketed into the project's central file.

5.2 Field Notebooks

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 Field Forms

All Tetra Tech NUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs contingent upon client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOP.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample Log Sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. A log sheet must be completed for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source electronically generated in-house, or are supplied from the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The Custody seal is an adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Field Analytical Log Sheets are used to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

A Groundwater Level Measurement Sheet must be filled out for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. The Pumping Test Data Sheet facilitates this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be laid out in advance.

5.3.2.3 Packer Test Report Form

A Packer Test Report Form must be completed for each well upon which a packer test is conducted.

5.3.2.4 Boring Log

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring, or Boring Log is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a PID or FID), these readings must be entered on the boring log at the appropriate depth. The "Remarks" column can be used to subsequently enter the laboratory sample number, the concentration of key analytical results, or other pertinent information. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

A Monitoring Well Construction Details Form must be completed for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation, or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

~~5.3.2.7 Miscellaneous Monitoring Well Forms~~

Monitoring Well Materials Certificate of Conformance should be used as the project directs to document all materials utilized during each monitoring well installation.

The Monitoring Well Development Record should be used as the project directs to document all well development activities.

5.3.2.8 Miscellaneous Field Forms - QA and Checklists

Container Sample and Inspection Sheet should be used as the project directs each time a container (drum, tank, etc.) is sampled and/or inspected.

QA Sample Log Sheet should be used at the project directs each time a QA sample is colleted, such as Rinsate Blank, Source Blank, etc.

Field Task Modification Request (FTMR) will be prepared for all deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Copies of all FTMRs will be maintained with the onsite planning documents and originals will be placed in the final evidence file.

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring or test equipment is necessary to assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device used in the field; entries must be made for each day the equipment is used or in accordance with the manufacturer's recommendations.

5.4 Field Reports

The primary means of recording onsite activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation, but are not easily useful for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain onsite for extended periods of time and are thus not accessible for timely review by project management.

5.4.1 Daily Activities Report

To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.

5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DAR reports are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the Project Manager.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

It should be noted that in addition to summaries described herein, other summary reports may also be contractually required.

All Tetra Tech NUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS

Groundwater Sample Log Sheet
Surface Water Sample Log Sheet
Soil/Sediment Sample Log Sheet
Container Sample and Inspection Sheet
Geochemical Parameters (Natural Attenuation)
Groundwater Level Measurement Sheet
Pumping Test Data Sheet
Packer Test Report Form
Boring Log
Monitoring Well Construction Bedrock Flush Mount
Monitoring Well Construction Bedrock Open Hole
Monitoring Well Construction Bedrock Stick Up
Monitoring Well Construction Confining Layer
Monitoring Well Construction Overburden Flush Mount
Monitoring Well Construction Overburden Stick Up
Test Pit Log
Monitoring Well Materials Certificate of Conformance
Monitoring Well Development Record

Daily Activities Record
Field Task Modification Request
Hydraulic Conductivity Test Data Sheet
Low Flow Purge Data Sheet
QA Sample Log Sheet
Equipment Calibration Log
Field Project Daily Activities Checklist
Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____
PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

_____

Field Operations Leader

ATTACHMENT B

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** **Site:** **Location:**	
Sample No:		**Matrix:**
Date:	**Time:**	**Preserve:**
Analysis:		
Sampled by:	**Laboratory:**	

ATTACHMENT C

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF ___

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

CONTAINER TYPE PLASTIC (P) or GLASS (G)
PRESERVATIVE USED
TYPE OF ANALYSIS

DATE YEAR	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS									COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME
COMMENTS					

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R
FORM NO. TtNUS-001

ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	**CUSTODY SEAL**
Date	**Date**
Signature	**Signature**

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1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The objective/purpose of this SOP is intended to protect site personnel, general public, and the sample integrity through the prevention of cross contamination onto unaffected persons or areas. It is further intended through this procedure to provide guidelines regarding the appropriate procedures to be followed when decontaminating drilling equipment, monitoring well materials, chemical sampling equipment and field analytical equipment.

2.0 SCOPE

This procedure applies to all equipment including drilling equipment, heavy equipment, monitoring well materials, as well as chemical sampling and field analytical equipment decontamination that may be used to provide access/acquire environmental samples. Where technologically and economically feasible, single use sealed disposable equipment will be employed to minimize the potential for cross contamination. This procedure also provides general reference information on the control of contaminated materials.

3.0 GLOSSARY

Acid - For decontamination of equipment when sampling for trace levels of inorganics, a 10% solution of nitric acid in deionized water should be used. Due to the leaching ability of nitric acid, it should not be used on stainless steel.

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - Is a solution selected/identified within the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Deionized water is tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet CAP and NCCLS specifications for reagent grade, Type I water.

Potable Water - Tap water used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Employs high pressure pumps and nozzle configuration to create a high pressure spray of potable water. High pressure spray is employed to remove solids.

Solvent - The solvent of choice is pesticide-grade Isopropanol. Use of other solvents (methanol, acetone, pesticide-grade hexane, or petroleum ether) may be required for particular projects or for a particular purpose (e.g. for the removal of concentrated waste) and must be justified in the project planning documents. As an example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - This method employs a high pressure spray of heated potable water. This method through the application of heat provides for the removal of various organic/inorganic compounds.

4.0 RESPONSIBILITIES

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved Standards Operating Procedures or as otherwise dictated by the approved project plan(s).

Site Health and Safety Officer (SHSO) - The SHSO exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on-site (as part of the equipment inspection), leaving the site, moving between locations are required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Failure to meet these objectives are sufficient to restrict equipment from entering the site/exiting the site/ or moving to a new location on the site until the objectives are successfully completed.

5.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or the isolation of contaminants. In order to accomplish this activity a level of preparation is required. This includes site preparation, equipment selection, and evaluation of the process. Site contaminant types, concentrations, media types, are primary drivers in the selection of the types of decontamination as well as where it will be conducted. For purposes of this SOP discussion will be provided concerning general environmental investigation procedures.

The decontamination processes are typically employed at:

- Temporary Decontamination Pads/Facilities
- Sample Locations
- Centralized Decontamination Pad/Facilities
- Combination of some or all of the above

The following discussion represents recommended site preparation in support of the decontamination process.

5.1 Decontamination Design/Constructions Considerations

5.1.1 Temporary Decontamination Pads

Temporary decontamination pads are constructed at satellite locations in support of temporary work sites. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soils generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations

- Site Location – The site selected should be within a reasonable distance from the work site but should avoid:

  - Pedestrian/Vehicle thoroughfares
  - Areas where control/custody cannot be maintained
  - Areas where a potential releases may be compounded through access to storm water transport systems, streams or other potentially sensitive areas.
  - Areas potentially contaminated.

- Pad – The pad should be constructed to provide the following characteristics

  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination.

  - Slope – An adequate slope will be constructed to permit the collection of the water and potentially contaminated soils within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks.

  - Sidewalls – The sidewalls should be a minimum of 6-inches in height to provide adequate containment for wash waters and soils. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls maybe constructed of wood, inflatables, sand bags, etc. to permit containment.

  - Liner – Depending on the types of equipment and the decontamination method the liner should be of sufficient thickness to provide a puncture resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. Achieving the desired thickness maybe achieved through layering lighter constructed materials. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner a light coating of sand maybe applied to provide traction as necessary.

  - Wash/drying Racks – Auger flights, drill/drive rods require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process. A minimum ground clearance of 2-feet is recommended.

  - Maintenance – The work area should be periodically cleared of standing water, soils, and debris. This action will aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross contamination. Hoses should be gathered when not in use to eliminate potential tripping hazards.

5.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and direct push activities decontamination of drive rods, Macro Core Samplers, split spoons, etc. are typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected media. Drying racks will be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/re-use.

5.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations sampling devices such as trowels, pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition.

5.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

5.2.1 Monitoring Well Sampling Equipment

5.2.1.1 Groundwater sampling pumps – This includes pumps inserted into the monitoring well such as Bladder pumps, Whale pumps, Redi-Flo, reusable bailers, etc.

1) Evacuate to the extent possible, any purge water within the pump.

2) Scrub using soap and water and/or steam clean the outside of the pump and tubing, where applicable.

3) Insert the pump and tubing into a clean container of soapy water. Pump a sufficient amount of soapy water through the pump to flush any residual purge water. Once flushed, circulate soapy water through the pump to ensure the internal components are thoroughly flushed.

4) Remove the pump and tubing from the container, rinse external components using tap water. Insert the pump and tubing into a clean container of tap water. Pump a sufficient amount of tap water through the pump to evacuate all of the soapy water (until clear).

5) Rinse equipment with pesticide grade isopropanol

6) Repeat item #4 using deionized water through the hose to flush out the tap water and solvent residue as applicable*.

7) Drain residual deionized water to the extent possible, allow components to air dry.

8) Wrap pump in aluminum foil or a clear clean plastic bag for storage.

5.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing with the extracted tape and probe with deionized water and wiping the surface of the extracted tape is acceptable. However, periodic full decontamination should be conducted as indicated below.

* - The solvent should be employed when samples contain oil, grease, PAHs, PCBs, and other hard to remove materials. If these are not of primary concern, the solvent step may be omitted. In addition, do not rinse PE, PVC, and associated tubing with solvents.

1) Wash with soap and water
2) Rinse with tap water
3) Rinse with deionized water

Note: In situations where oil, grease, free product, other hard to remove materials are encountered probes and exposed tapes should be washed in hot soapy water.

5.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) should be cleaned per manufacturer's instructions. This generally includes wiping down the sensor housing and rinsing with tap and deionized water.

Coolers/Shipping Containers employed to ship samples are received from the lab in a variety of conditions from marginal to extremely poor. Coolers should be evaluated prior to use for

- Structural integrity – Coolers missing handles or having breaks within the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples will not be attempted and request a replacement unit.

- Cleanliness – As per protocol only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or associated with noticeable odors it should be decontaminated prior to use.

  1) Wash with soap and water
  2) Rinse with tap water
  3) Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and notify the laboratory to provide a replacement unit.

5.2.2 Down-Hole Drilling Equipment

This includes any portion of the drill rig that is over the borehole including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. This procedure is to be employed prior to initiating the drilling/sampling activity, then between locations.

1) Remove all soils to the extent possible using shovels, scrapers, etc. to remove loose soils.
2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.
3) Rinse with tap water.
4) Rinse equipment with pesticide grade isopropanol
5) To the extent possible allow components to air dry.
6) Wrap or cover equipment in clear plastic until it is time to be used.

5.2.3 Soil/Sediment Sampling Equipment

This consists of soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1) Remove all soils to the extent possible.

2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.

3) Rinse with tap water.

4) Rinse equipment with pesticide grade isopropanol

5) Rinse with deionized water

6) To the extent possible allow components to air dry.

7) If the device is to be used immediately, screen with a PID/FID to insure all solvents (if they were used) and trace contaminants have been adequately removed.

8) Once these devices have been dried wrap in aluminum foil for storage until it is time to be used.

5.3 Contact Waste/Materials

During the course of field investigations disposable/single use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.) broken sample containers.

With the exception of the broken glass, single use articles should be cleaned (washed and rinsed) of visible materials and disposed of as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned should be containerized for disposal in accordance with applicable federal state and local regulations.

5.3.1 Decontamination Solutions

All waste decontamination solutions and rinses must be assumed to contain the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. The waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility. These containers must be appropriately labeled.

5.4 Decontamination Evaluation

Determining the effectiveness of the decontamination process will be accomplished in the following manner

- Visual Evaluation – A visual evaluation will be conducted to insure the removal of particulate matter. This will be done to insure that the washing/rinsing process is working as intended.

- Instrument Screening – A PID and/or an FID should be used to evaluate the presence of the contaminants or solvents used in the cleaning process. The air intake of the instrument should be passed over the article to be evaluated. A positive detection requires a repeat the decontamination process. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instruments capabilities.

- Rinsate Blanks – It is recommended that Rinsate samples be collected to
  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.
  - Single use disposable equipment – The number of samples should represent different types of equipment as well as different Lot Numbers of single use articles.

The collection and the frequency of collection of rinsate samples are as follows:

- Per decontamination method
- Per disposable article/Batch number of disposable articles

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and in an effort to avoid using a contaminated batch of single use articles. It is recommended that a follow up sample be collected during the execution of the project to insure those conditions do not change. Lastly, rinsate samples collection may be driven by types of and/or contaminant levels. Hard to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

APPENDIX C

LABORATORY STANDARD OPERATING PROCEDURES AND CERTIFICATIONS

LABORATORY ACCREDITATION BUREAU

Certificate of Accreditation

ISO/IEC 17025:2005 ***Certificate Number L2247***

MITKEM

175 Metro Center Blvd
Warwick, RI 02886

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: April 1, 2013

R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 1st of April 2010

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements
Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2247

Scope of Accreditation For MITKEM

175 Metro Center Blvd.
Warwick, RI 02886
Sharyn Lawler
401-732-3400

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to MITKEM to perform the following tests:

Accreditation granted through: April 1, 2013

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethene
GC/MS	EPA 8260C	1,1-Dichloropropene
GC/MS	EPA 8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260C	1,2-Dibromoethane
GC/MS	EPA 8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260C	1,2-Dichloroethane
GC/MS	EPA 8260C	1,2-Dichloropropane
GC/MS	EPA 8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260C	1,3-Dichloropropane
GC/MS	EPA 8260C	1,4-Dichlorobenzene

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8260C	1-Chlorohexane
GC/MS	EPA 8260C	2,2-Dichloropropane
GC/MS	EPA 8260C	2-Butanone
GC/MS	EPA 8260C	2-Chlorotoluene
GC/MS	EPA 8260C	2-Hexanone
GC/MS	EPA 8260C	4-Chlorotoluene
GC/MS	EPA 8260C	4-Isopropyltoluene
GC/MS	EPA 8260C	4-Methyl-2-pentanone
GC/MS	EPA 8260C	Acetone
GC/MS	EPA 8260C	Acetonitrile
GC/MS	EPA 8260C	Acrolein
GC/MS	EPA 8260C	Acrylonitrile
GC/MS	EPA 8260C	Allyl Chloride
GC/MS	EPA 8260C	Benzene
GC/MS	EPA 8260C	Bromobenzene
GC/MS	EPA 8260C	Bromochloromethane
GC/MS	EPA 8260C	Bromodichloromethane
GC/MS	EPA 8260C	Bromoform
GC/MS	EPA 8260C	Bromomethane
GC/MS	EPA 8260C	Carbon disulfide
GC/MS	EPA 8260C	Carbon tetrachloride
GC/MS	EPA 8260C	Chlorobenzene
GC/MS	EPA 8260C	Chloroethane
GC/MS	EPA 8260C	Chloroform
GC/MS	EPA 8260C	Chloromethane
GC/MS	EPA 8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260C	Cyclohexane
GC/MS	EPA 8260C	Dibromochloromethane
GC/MS	EPA 8260C	Dibromomethane
GC/MS	EPA 8260C	Dichlorodifluoromethane
GC/MS	EPA 8260C	Diethyl Ether
GC/MS	EPA 8260C	Diisopropyl ether
GC/MS	EPA 8260C	Ethanol
GC/MS	EPA 8260C	Ethylbenzene
GC/MS	EPA 8260C	Ethyl methacrylate
GC/MS	EPA 8260C	Ethyl tert-butyl ether
GC/MS	EPA 8260C	Hexachlorobutadiene
GC/MS	EPA 8260C	Hexachloroethane
GC/MS	EPA 8260C	Iodomethane
GC/MS	EPA 8260C	Isobutyl alcohol
GC/MS	EPA 8260C	Isopropylbenzene
GC/MS	EPA 8260C	m,p-Xylene
GC/MS	EPA 8260C	Methacrylonitrile
GC/MS	EPA 8260C	Methyl acetate

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8260C	Methylcyclohexane
GC/MS	EPA 8260C	Methyl methacrylate
GC/MS	EPA 8260C	Methyl tert-butyl ether
GC/MS	EPA 8260C	Methylene chloride
GC/MS	EPA 8260C	n-Butylbenzene
GC/MS	EPA 8260C	n-Propylbenzene
GC/MS	EPA 8260C	Naphthalene
GC/MS	EPA 8260C	o-Xylene
GC/MS	EPA 8260C	Proprionitrile
GC/MS	EPA 8260C	sec-Butylbenzene
GC/MS	EPA 8260C	Styrene
GC/MS	EPA 8260C	tert-Amyl Methyl ether
GC/MS	EPA 8260C	tert-Butyl alcohol
GC/MS	EPA 8260C	tert-Butylbenzene
GC/MS	EPA 8260C	Tetrachloroethene
GC/MS	EPA 8260C	Tetrahydrofuran
GC/MS	EPA 8260C	Toluene
GC/MS	EPA 8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260C	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260C	Trichloroethene
GC/MS	EPA 8260C	Trichlorofluoromethane
GC/MS	EPA 8260C	Vinyl acetate
GC/MS	EPA 8260C	Vinyl chloride
GC/MS	EPA 8260C	Xylene (Total)
GC/MS	EPA 8270D	1,1´-Biphenyl
GC/MS	EPA 8270D	Benzaldehyde
GC/MS	EPA 8270D	Caprolactam
GC/MS	EPA 8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dioxane
GC/MS	EPA 8270D	1-Methylnaphthalene
GC/MS	EPA 8270D	2,2´-oxybis(1-Chloropropane)
GC/MS	EPA 8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270D	2,4-Dichlorophenol
GC/MS	EPA 8270D	2,4-Dimethylphenol
GC/MS	EPA 8270D	2,4-Dinitrophenol
GC/MS	EPA 8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270D	2-Chloronaphthalene
GC/MS	EPA 8270D	2-Chlorophenol
GC/MS	EPA 8270D	2-Methylnaphthalene

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8270D	2-Methylphenol
GC/MS	EPA 8270D	2-Nitroaniline
GC/MS	EPA 8270D	2-Nitrophenol
GC/MS	EPA 8270D	3,3´-Dichlorobenzidine
GC/MS	EPA 8270D	3-Nitroaniline
GC/MS	EPA 8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270D	4-Bromophenyl-phenylether
GC/MS	EPA 8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270D	4-Chloroaniline
GC/MS	EPA 8270D	4-Chlorophenyl-phenylether
GC/MS	EPA 8270D	4-Methylphenol
GC/MS	EPA 8270D	4-Nitroaniline
GC/MS	EPA 8270D	4-Nitrophenol
GC/MS	EPA 8270D	Acenaphthene
GC/MS	EPA 8270D	Acenaphthylene
GC/MS	EPA 8270D	Aniline
GC/MS	EPA 8270D	Anthracene
GC/MS	EPA 8270D	Atrazine
GC/MS	EPA 8270D	Azobenzene
GC/MS	EPA 8270D	Benzidine
GC/MS	EPA 8270D	Benzyl Alcohol
GC/MS	EPA 8270D	Benzo(a)anthracene
GC/MS	EPA 8270D	Benzo(a)pyrene
GC/MS	EPA 8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270D	Benzo(g,h,i)perylene
GC/MS	EPA 8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270D	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270D	Bis(2-chloroethyl)ether
GC/MS	EPA 8270D	Bis(2-ethylhexyl)phthalate
GC/MS	EPA 8270D	Butylbenzylphthalate
GC/MS	EPA 8270D	Carbazole
GC/MS	EPA 8270D	Chrysene
GC/MS	EPA 8270D	Di-n-butylphthalate
GC/MS	EPA 8270D	Dibenzofuran
GC/MS	EPA 8270D	Diethylphthalate
GC/MS	EPA 8270D	Dimethylphthalate
GC/MS	EPA 8270D	Di-n-octylphthalate
GC/MS	EPA 8270D	Dibenzo(a,h)anthracene
GC/MS	EPA 8270D	Fluoranthene
GC/MS	EPA 8270D	Fluorene
GC/MS	EPA 8270D	Hexachlorobenzene
GC/MS	EPA 8270D	Hexachlorobutadiene
GC/MS	EPA 8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270D	Hexachloroethane
GC/MS	EPA 8270D	Indeno(1,2,3-cd)pyrene

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8270D	Isophorone
GC/MS	EPA 8270D	N-Nitroso-di-n-propylamine
GC/MS	EPA 8270D	Nitrobenzene
GC/MS	EPA 8270D	Pentachlorophenol
GC/MS	EPA 8270D	N-Nitrosodimethylamine
GC/MS	EPA 8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270D	Naphthalene
GC/MS	EPA 8270D	Phenanthrene
GC/MS	EPA 8270D	Phenol
GC/MS	EPA 8270D	Pyrene
GC/MS	EPA 8270D	Pyridine
GC/ECD	EPA 8081B	4,4´-DDD
GC/ECD	EPA 8081B	4,4´-DDE
GC/ECD	EPA 8081B	4,4´-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC
GC/ECD	EPA 8081B	alpha-Chlordane
GC/ECD	EPA 8081B	beta-BHC
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane)
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Toxaphene
GC/ECD	EPA 8081B	Chlordane (technical)
GC/ECD	EPA 8082A	Aroclor-1016
GC/ECD	EPA 8082A	Aroclor-1221
GC/ECD	EPA 8082A	Aroclor-1232
GC/ECD	EPA 8082A	Aroclor-1242
GC/ECD	EPA 8082A	Aroclor-1248
GC/ECD	EPA 8082A	Aroclor-1254
GC/ECD	EPA 8082A	Aroclor-1260
GC/ECD	EPA 8082A	Aroclor-1262
GC/ECD	EPA 8082A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D

Non-Potable Water

Technology	Method	Analyte
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/FID	EPA 8015D	Diesel Range Organics
GC/FID	EPA 8015D	Gasoline Range Organics
ICP/AES	EPA 6010C	Aluminum
ICP/AES	EPA 6010C	Antimony
ICP/AES	EPA 6010C	Arsenic
ICP/AES	EPA 6010C	Barium
ICP/AES	EPA 6010C	Beryllium
ICP/AES	EPA 6010C	Boron
ICP/AES	EPA 6010C	Cadmium
ICP/AES	EPA 6010C	Calcium
ICP/AES	EPA 6010C	Chromium
ICP/AES	EPA 6010C	Cobalt
ICP/AES	EPA 6010C	Copper
ICP/AES	EPA 6010C	Iron
ICP/AES	EPA 6010C	Lead
ICP/AES	EPA 6010C	Magnesium
ICP/AES	EPA 6010C	Manganese
ICP/AES	EPA 6010C	Molybdenum
ICP/AES	EPA 6010C	Nickel
ICP/AES	EPA 6010C	Potassium
ICP/AES	EPA 6010C	Selenium
ICP/AES	EPA 6010C	Silver
ICP/AES	EPA 6010C	Sodium
ICP/AES	EPA 6010C	Thallium
ICP/AES	EPA 6010C	Tin
ICP/AES	EPA 6010C	Vanadium
ICP/AES	EPA 6010C	Zinc
ICP/AES	SM 2340 B	Hardness, Ca/Mg (As CaCO3) BY CALCULATION
ICP/MS	EPA 6020A	Aluminum
ICP/MS	EPA 6020A	Antimony
ICP/MS	EPA 6020A	Arsenic
ICP/MS	EPA 6020A	Barium
ICP/MS	EPA 6020A	Beryllium
ICP/MS	EPA 6020A	Cadmium
ICP/MS	EPA 6020A	Calcium
ICP/MS	EPA 6020A	Chromium
ICP/MS	EPA 6020A	Cobalt
ICP/MS	EPA 6020A	Copper

Non-Potable Water

Technology	Method	Analyte
ICP/MS	EPA 6020A	Iron
ICP/MS	EPA 6020A	Lead
ICP/MS	EPA 6020A	Magnesium
ICP/MS	EPA 6020A	Manganese
ICP/MS	EPA 6020A	Nickel
ICP/MS	EPA 6020A	Potassium
ICP/MS	EPA 6020A	Selenium
ICP/MS	EPA 6020A	Silver
ICP/MS	EPA 6020A	Sodium
ICP/MS	EPA 6020A	Thallium
ICP/MS	EPA 6020A	Vanadium
ICP/MS	EPA 6020A	Zinc
CVAA	EPA 7470A	Mercury
FIA	EPA 9012B	Total Cyanide
IC	EPA 9056A	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrogen, Nitrate (As N)
IC	EPA 9056A	Nitrogen, Nitrite (As N)
FIA	EPA 353.2	Nitrogen, Nitrate-Nitrite
IC	EPA 9056A	ortho-Phosphate (As P)
UV/VIS	SM 4500 P B(5)+E 18th ED	Total Phosphorus
IC	EPA 9056A	Sulfate
IC	EPA 300.0 mod.	Acetic Acid
IC	EPA 300.0 mod.	Butyric Acid
IC	EPA 300.0 mod.	Lactic Acid
IC	EPA 300.0 mod.	Propionic Acid
IC	EPA 300.0 mod.	Pyruvic Acid
UV/VIS	SM 4500 S2- D 20th ED	Sulfide
combustion/IR	EPA 9060A	Organic Carbon, Total
UV/VIS	SM 3500 Cr D 18th ED	Chromium, Hexavalent
Pensky-Marten	EPA 1010	Ignitability
pH meter	SM 4500 H+B 18th ED	pH
UV/VIS	SM 4500 NH3 B,C 18th ED	Ammonia-N
UV/VIS	SM 4500 N Org C 20th ED	TKN-N
Titration	SM 2320 B 20th ED	Alkalinity, Total (As CaCO3)
Gravimetric	SM 2540 C 20th ED	Total Dissolved Solids
Gravimetric	SM 2540 D 20th ED	Total Suspended Solids
Gravimetric	EPA 1664A	Oil & Grease, Total Recoverable
Conductivity Meter	EPA 120.1	Specific Conductance
UV/VIS	SM 5220 D 20th ED	Chemical Oxygen Demand
UV/VIS	SM 3500 Fe B 20th ED	Ferrous Iron
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/FID	RSK-175	Methane

Non-Potable Water

Preparation	Method	Type
Organic Preparation	EPA 3510C	Separatory Funnel
Organic Preparation	EPA 3520C	Continuous Liquid Liquid
Inorganic Preparation	EPA 3005A	Hotblock
Inorganic Preparation	EPA 3010A	Hotblock
Volatile Organic Preparation	EPA 5030B	Purge and Trap

Solid and Chemical Waste

Technology	Method	Analyte
CVAA	EPA 7471B	Mercury
FIA	EPA 9012B	Total Cyanide
Titration	WALKLEY BLACK	Organic Carbon, Total
Combustion/IR	EPA 9060A	Organic Carbon, Total
Combustion/IR	Lloyd Kahn	Organic Carbon, Total
UV/VIS	EPA 7196A	Chromium, Hexavalent
Oven	ASTM D2216	Percent moisture
pH meter	EPA 9045C	pH
ICP/AES	EPA 6010C	Aluminum
ICP/AES	EPA 6010C	Antimony
ICP/AES	EPA 6010C	Arsenic
ICP/AES	EPA 6010C	Barium
ICP/AES	EPA 6010C	Beryllium
ICP/AES	EPA 6010C	Boron
ICP/AES	EPA 6010C	Cadmium
ICP/AES	EPA 6010C	Calcium
ICP/AES	EPA 6010C	Chromium
ICP/AES	EPA 6010C	Cobalt
ICP/AES	EPA 6010C	Copper
ICP/AES	EPA 6010C	Iron
ICP/AES	EPA 6010C	Lead
ICP/AES	EPA 6010C	Magnesium
ICP/AES	EPA 6010C	Manganese
ICP/AES	EPA 6010C	Molybdenum
ICP/AES	EPA 6010C	Nickel
ICP/AES	EPA 6010C	Potassium
ICP/AES	EPA 6010C	Selenium
ICP/AES	EPA 6010C	Silver
ICP/AES	EPA 6010C	Sodium
ICP/AES	EPA 6010C	Thallium
ICP/AES	EPA 6010C	Tin
ICP/AES	EPA 6010C	Vanadium
ICP/AES	EPA 6010C	Zinc
ICP/MS	EPA 6020A	Aluminum
ICP/MS	EPA 6020A	Antimony

Solid and Chemical Waste

Technology	Method	Analyte
ICP/MS	EPA 6020A	Arsenic
ICP/MS	EPA 6020A	Barium
ICP/MS	EPA 6020A	Beryllium
ICP/MS	EPA 6020A	Cadmium
ICP/MS	EPA 6020A	Calcium
ICP/MS	EPA 6020A	Chromium
ICP/MS	EPA 6020A	Cobalt
ICP/MS	EPA 6020A	Copper
ICP/MS	EPA 6020A	Iron
ICP/MS	EPA 6020A	Lead
ICP/MS	EPA 6020A	Magnesium
ICP/MS	EPA 6020A	Manganese
ICP/MS	EPA 6020A	Nickel
ICP/MS	EPA 6020A	Potassium
ICP/MS	EPA 6020A	Selenium
ICP/MS	EPA 6020A	Silver
ICP/MS	EPA 6020A	Sodium
ICP/MS	EPA 6020A	Thallium
ICP/MS	EPA 6020A	Vanadium
ICP/MS	EPA 6020A	Zinc
GC/FID	EPA 8015D	Diesel Range Organics
GC/FID	EPA 8015D	Gasoline Range Organics
GC/ECD	EPA 8082A	Aroclor-1016
GC/ECD	EPA 8082A	Aroclor-1221
GC/ECD	EPA 8082A	Aroclor-1232
GC/ECD	EPA 8082A	Aroclor-1242
GC/ECD	EPA 8082A	Aroclor-1248
GC/ECD	EPA 8082A	Aroclor-1254
GC/ECD	EPA 8082A	Aroclor-1260
GC/ECD	EPA 8082A	Aroclor-1262
GC/ECD	EPA 8082A	Aroclor-1268
GC/ECD	EPA 8081B	4,4´-DDD
GC/ECD	EPA 8081B	4,4´-DDE
GC/ECD	EPA 8081B	4,4´-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC
GC/ECD	EPA 8081B	alpha-Chlordane
GC/ECD	EPA 8081B	beta-BHC
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde

Solid and Chemical Waste

Technology	Method	Analyte
GC/ECD	EPA 8081B	Endrin ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane)
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Toxaphene
GC/ECD	EPA 8081B	Chlordane (technical)
GC/MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethene
GC/MS	EPA 8260C	1,1-Dichloropropene
GC/MS	EPA 8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260C	1,2-Dibromoethane
GC/MS	EPA 8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260C	1,2-Dichloroethane
GC/MS	EPA 8260C	1,2-Dichloropropane
GC/MS	EPA 8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260C	1,3-Dichloropropane
GC/MS	EPA 8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260C	1,4-Dioxane
GC/MS	EPA 8260C	1-Chlorohexane
GC/MS	EPA 8260C	2,2-Dichloropropane
GC/MS	EPA 8260C	2-Butanone
GC/MS	EPA 8260C	2-Chlorotoluene
GC/MS	EPA 8260C	2-Hexanone
GC/MS	EPA 8260C	4-Chlorotoluene
GC/MS	EPA 8260C	4-Isopropyltoluene
GC/MS	EPA 8260C	4-Methyl-2-pentanone
GC/MS	EPA 8260C	Acetone
GC/MS	EPA 8260C	Acetonitrile
GC/MS	EPA 8260C	Acrolein
GC/MS	EPA 8260C	Acrylonitrile
GC/MS	EPA 8260C	Allyl Chloride
GC/MS	EPA 8260C	Benzene
GC/MS	EPA 8260C	Bromobenzene

Solid and Chemical Waste

Technology	Method	Analyte
GC/MS	EPA 8260C	Bromochloromethane
GC/MS	EPA 8260C	Bromodichloromethane
GC/MS	EPA 8260C	Bromoform
GC/MS	EPA 8260C	Bromomethane
GC/MS	EPA 8260C	Carbon disulfide
GC/MS	EPA 8260C	Carbon tetrachloride
GC/MS	EPA 8260C	Chlorobenzene
GC/MS	EPA 8260C	Chloroethane
GC/MS	EPA 8260C	Chloroform
GC/MS	EPA 8260C	Chloromethane
GC/MS	EPA 8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260C	Cyclohexane
GC/MS	EPA 8260C	Dibromochloromethane
GC/MS	EPA 8260C	Dibromomethane
GC/MS	EPA 8260C	Dichlorodifluoromethane
GC/MS	EPA 8260C	Diethyl Ether
GC/MS	EPA 8260C	Diisopropyl ether
GC/MS	EPA 8260C	Ethanol
GC/MS	EPA 8260C	Ethylbenzene
GC/MS	EPA 8260C	Ethyl methacrylate
GC/MS	EPA 8260C	Ethyl tert-butyl ether
GC/MS	EPA 8260C	Hexachlorobutadiene
GC/MS	EPA 8260C	Hexachloroethane
GC/MS	EPA 8260C	Iodomethane
GC/MS	EPA 8260C	Isobutyl alcohol
GC/MS	EPA 8260C	Isopropylbenzene
GC/MS	EPA 8260C	m,p-Xylene
GC/MS	EPA 8260C	Methacrylonitrile
GC/MS	EPA 8260C	Methyl acetate
GC/MS	EPA 8260C	Methylcyclohexane
GC/MS	EPA 8260C	Methyl methacrylate
GC/MS	EPA 8260C	Methyl tert-butyl ether
GC/MS	EPA 8260C	Methylene chloride
GC/MS	EPA 8260C	n-Butylbenzene
GC/MS	EPA 8260C	n-Propylbenzene
GC/MS	EPA 8260C	Naphthalene
GC/MS	EPA 8260C	o-Xylene
GC/MS	EPA 8260C	Proprionitrile
GC/MS	EPA 8260C	sec-Butylbenzene
GC/MS	EPA 8260C	Styrene
GC/MS	EPA 8260C	tert-Amyl Methyl ether
GC/MS	EPA 8260C	tert-Butyl alcohol
GC/MS	EPA 8260C	tert-Butylbenzene
GC/MS	EPA 8260C	Tetrachloroethene

Solid and Chemical Waste

Technology	Method	Analyte
GC/MS	EPA 8260C	Tetrahydrofuran
GC/MS	EPA 8260C	Toluene
GC/MS	EPA 8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260C	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260C	Trichloroethene
GC/MS	EPA 8260C	Trichlorofluoromethane
GC/MS	EPA 8260C	Vinyl acetate
GC/MS	EPA 8260C	Vinyl chloride
GC/MS	EPA 8260C	Xylene (Total)
GC/MS	EPA 8270D	1,1´-Biphenyl
GC/MS	EPA 8270D	Benzaldehyde
GC/MS	EPA 8270D	Caprolactam
GC/MS	EPA 8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270D	1-Methylnaphthalene
GC/MS	EPA 8270D	2,2´-oxybis(1-Chloropropane)
GC/MS	EPA 8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270D	2,4-Dichlorophenol
GC/MS	EPA 8270D	2,4-Dimethylphenol
GC/MS	EPA 8270D	2,4-Dinitrophenol
GC/MS	EPA 8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270D	2-Chloronaphthalene
GC/MS	EPA 8270D	2-Chlorophenol
GC/MS	EPA 8270D	2-Methylnaphthalene
GC/MS	EPA 8270D	2-Methylphenol
GC/MS	EPA 8270D	2-Nitroaniline
GC/MS	EPA 8270D	2-Nitrophenol
GC/MS	EPA 8270D	3,3´-Dichlorobenzidine
GC/MS	EPA 8270D	3-Nitroaniline
GC/MS	EPA 8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270D	4-Bromophenyl-phenylether
GC/MS	EPA 8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270D	4-Chloroaniline
GC/MS	EPA 8270D	4-Chlorophenyl-phenylether
GC/MS	EPA 8270D	4-Methylphenol
GC/MS	EPA 8270D	4-Nitroaniline
GC/MS	EPA 8270D	4-Nitrophenol
GC/MS	EPA 8270D	Acenaphthene
GC/MS	EPA 8270D	Acenaphthylene
GC/MS	EPA 8270D	Aniline

Solid and Chemical Waste

Technology	Method	Analyte
GC/MS	EPA 8270D	Anthracene
GC/MS	EPA 8270D	Atrazine
GC/MS	EPA 8270D	Azobenzene
GC/MS	EPA 8270D	Benzyl Alcohol
GC/MS	EPA 8270D	Benzo(a)anthracene
GC/MS	EPA 8270D	Benzo(a)pyrene
GC/MS	EPA 8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270D	Benzo(g,h,i)perylene
GC/MS	EPA 8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270D	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270D	Bis(2-chloroethyl)ether
GC/MS	EPA 8270D	Bis(2-ethylhexyl)phthalate
GC/MS	EPA 8270D	Butylbenzylphthalate
GC/MS	EPA 8270D	Carbazole
GC/MS	EPA 8270D	Chrysene
GC/MS	EPA 8270D	Di-n-butylphthalate
GC/MS	EPA 8270D	Dibenzofuran
GC/MS	EPA 8270D	Diethylphthalate
GC/MS	EPA 8270D	Dimethylphthalate
GC/MS	EPA 8270D	Di-n-octylphthalate
GC/MS	EPA 8270D	Dibenzo(a,h)anthracene
GC/MS	EPA 8270D	Fluoranthene
GC/MS	EPA 8270D	Fluorene
GC/MS	EPA 8270D	Hexachlorobenzene
GC/MS	EPA 8270D	Hexachlorobutadiene
GC/MS	EPA 8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270D	Hexachloroethane
GC/MS	EPA 8270D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D	Isophorone
GC/MS	EPA 8270D	N-Nitroso-di-n-propylamine
GC/MS	EPA 8270D	Nitrobenzene
GC/MS	EPA 8270D	Pentachlorophenol
GC/MS	EPA 8270D	N-Nitrosodimethylamine
GC/MS	EPA 8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270D	Naphthalene
GC/MS	EPA 8270D	Phenanthrene
GC/MS	EPA 8270D	Phenol
GC/MS	EPA 8270D	Pyrene
UV/VIS	EPA 9031	Extractable Sulfides
Preparation	**Method**	**Type**
Organic Preparation	EPA 3550B	Sonication
Inorganics Preparation	EPA 3050B	Hotblock

LABORATORY ACCREDITATION BUREAU

Certificate # L2247

Solid and Chemical Waste		
Preparation	**Method**	**Type**
Volatile Organics Prepration	EPA 5035	Closed System Purge and Trap
Inorganics Preparation	EPA 3060A	Alkaline Digestion
Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
Preparation	ASTM D3987	Shake ext of solid waste with water

Notes:

1) This laboratory offers commercial testing service.

Approved By:_____ Date: April 6, 2010

R. Douglas Leonard
Chief Technical Officer

Issued: 04/01/10 Revised 04/06/10

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE
For

Logging Workorders and Samples into Omega ME.

SOP 20.0003

Rev. 4

	Signature	Date
QA Director:		11/9/09
Lab Director:		11/10/09

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE
For

Logging Workorders and Samples into Omega ME

Rev. 4

1. Scope and/or Applications

This Standard Operating Procedure (SOP) describes the procedures used to create workorders and log samples into Omega ME. Other steps in sample receipt and chain of custody documentation are described in separate SOP, Number 30.0003, entitled SOP for Sample Receipt, Storage and Tracking

2. Personnel Qualification and Responsibilities

Employees should have read and understood this SOP.

3. Summary of Procedure

This document assumes that a client and project profile has already been defined in Omega ME.

IMPORTANT: Omega ME generates unique sequential numbers (workorder numbers). Work order numbers consist of one alpha character followed by a four digit number YXXXX, in which Y is the alpha character indicating the number of years that the Omega LIMS System has been operational and XXXX is a sequential number for this workorder for the current year. These are assigned in order as samples are received at the laboratory. **It is essential that all new workorder numbers be assigned in sequence.** This number is assigned by the laboratory and is recorded on the chain of custody form **before** the samples are logged into Omega ME. The Omega ME workorder number and the Mitkem project number are the same. If care is not taken, the information logged into Omega ME workorder and the information in the Mitkem Project folder will not match.

4. Sample Preservation, Container, Handling and Storage

Not applicable

5. Interferences and Potential Problems

Not applicable

6. Equipment and Apparatus

Not applicable

7. Reagents

Not applicable

8. Procedure

8.1 CREATING A WORKORDER

8.1.1 From the main page of Omega ME click [WorkOrders] **Figure 1.**

8.1.2 The Omega ME - WorkOrders Form will open.

8.1.3 On the menu bar at the top of the screen click the [Add] button. This will add the next available unique WorkOrder number and clear the screen of any prior information.

On the screen click the down arrow next to the [Client ID] button to open a list of all of Mitkem's current clients. Instead of searching through the long list of clients type in the first letters of the client name to shorten the selection. Select the proper client. The client name and address will automatically appear in the Client Information box. **Figure 2.**

Choose the appropriate project by pressing the down arrow on the [Client Project] button, **Figure 3**. If the client provides a purchase number (PO) enter it in the box provided.

Enter the received date (date samples received by the lab). It is necessary to assign the workorder numbers in order by date samples received. The hardcopy due date [HC Due] is then filled in automatically by Omega based on the number of days (TAT HC/FAX) listed.

If a workorder requires a short turnaround time (**24, 48 or 72 hours, etc.**), click the checkmark for the [**Fax Results to Client**] box. The cursor will automatically move to the (**TAT HC/FX**) box which is the box on the right-hand side in which the turnaround time number will need to be entered: **24=1, 48=2,72=3**. When this is complete tab down to the box that is highlighted red which will exhibit the date the workorder is due. If the hardcopy due date differs from the fax due date, enter this information now.

For EPA workorders the client is **EPAINV.** The project (I,II,III,IV,V,VI,VII,VIII,IX,X, SOM_PE) is next selected by looking at the Scheduling Notification Form. This form identifies the case number, the Region the samples are from, the analyses required and turnaround time. Setting up an EPA workorder differs from a commercial workorder in that the Case Number and SDG fields must be completed from the Chain of Custody after protocol has been selected. In some cases, a particular Case will have samples for both water and soils. The soil samples and aqueous samples may be logged into separate Work Orders. The Mitkem project manager will provide this information. Some EPA / commercial projects are kept open for more than one day, so be careful to change the received date as the samples arrive at the lab in order to update the due date properly. Click the [Login] button to begin logging in the samples.

8.1.4 A new screen appears in order to log in the sample information. The fields that need to be completed include:

1. **Click on ADD Samp.** This will add the first sample to be logged in or the information entered into the other fields will not be saved. **Date Received:** Enter the date and time that the samples were received, **Figure 4.**
2. **Client Sample ID:** Type the client ID for each sample as it appears on the Chain of Custody. **Tag Number:** If the ID needs to be truncated because it is more than 10 characters long, type the truncated ID in the Tag Number field. Example: **SB29S-32-12302 = 29S3212302.**
3. **Collection Date:** Enter the date and time that the sample was collected, if a date and time are present. A date is necessary as this is used to keep track of hold times (for non-EPA work). The time of collection is not a necessity, unless the analysis has a 24-hour or 48-hour hold time.
4. **Matrix:** Select the down arrow and choose the appropriate matrix for the sample. If a solid matrix is chosen the PMOIST automatically appears to schedule a percent solids analysis.
5. **Bottle Type:** Choose the type of bottle for each sample fraction by clicking the down arrow and selecting the appropriate bottle.
6. **Containers:** Enter the total number of bottles received per sample.
7. **Storage/Status:** Type the location where the bottle will be stored in the Sample Receiving area. Most of the samples are stored in the walk-in (R1). VOA samples are stored in the refrigerators in the VOA lab. Aqueous metals are stored outside the walk-in cooler on a metal rack. ISM metals and cyanide samples must be stored in the walk-in. Metals samples may be stored at room temperature after digestion is completed.
8. **COC ID:** Some clients require this information to be included. The Mitkem project manager will notify the Receiving department when this needs to be done. The COC ID is required for SOM01.2. If there is more than one COC, associate the correct COC ID to the correct sample. To input the COC ID, go to the AFCEE tab and type in the COC ID in COC ID. **Figure 5**
9. **SA (CODE):** If the workorder requires the COC ID field to be completed, then this field also must be completed. Select whether the sample is a FB (field blank), TB

(trip blank), RB (rinsate blank), or EB (equipment blank). This information can be found on the COC. This field is found under the AFCEE tab

10. **Temp (C): The temperature of the cooler needs to be completed for SOM01.2. In EPA cases where there is more than one cooler received for a particular Case, the correct temperature needs to be associated with the correct samples.** This field is also found under the AFCEE tab.
11. When all the above information has been entered, the next step is to choose the correct **Test**. The tests for a particular project may be found by clicking on the Test column and using the drop down arrow. Most test methods will automatically associate a preparatory method. If no preparatory method appears, contact the project manager or IT department for assistance. There are only a few tests which do not have associated prep and all others MUST have preparation logged in.
12. Click ADD Frac if another fraction needs to be added. Fractions are added when different bottles with different preservatives are present. For example, a client sends in a sample for volatile, semivolatile, pesticides, metals and cyanide analysis. There will be an "A", a "B", a "C" and a "D" fraction. For example, they will be logged in as "A" for volatile, "B" for semivolatile and pesticides, "C" for metals and "D" for cyanide. This is due to each sample bottle being different and having different preservatives. **Figure 6**.
13. To add additional samples, click on the ADD Samp button. Enter the sample ID, date and time of collection, matrix, and type of bottle and storage location. Once all this info has been entered, choose the test. An easier method when logging in a number of samples with identical analyses is to use the Multilog button, Figure 6. Clicking the Multilog button automatically copies all information to the next sample except for Client ID, Date and Time of collection, and Tag Number. If multi-logging, type the client ID, date of collection and time of collection for each sample. Once this is done, click on POST. **Figure 7**.
14. If samples arrive with designated QC (duplicates and matrix spikes) then check the MS button on the right side of the screen after you have chosen the appropriate test. Be sure to change the number of bottles for this sample, as there will be more sample bottles. **Figure 8**
15. If specific tests are included on the Chain of Custody but the analysis is on hold, then check the "Sample on Hold" box. **Figure 9**
16. Comments should be added if warranted. For example, if there is limited sample volume, or the Chain-of-Custody indicates that the samples are dirty. Go to the Comments: and type in the necessary comment.
17. Labels are printed and affixed to the bottles. Labels may be printed by clicking on the Label button. Click over the samples in which labels need to be printed. Click the OK button and then the printer icon.
18. Once the labels have been printed, a work order needs to be printed. Back out to the work order page. Click on WO Rpt. Place the work order into the folder. **Figures 10 and 11**
19. A preliminary invoice will also need to be printed and placed into the folder. Click on the Reports button. Click on Base Report. Click on Clear All. Check on Invoice and then click on Preview Report and print the invoice. **Figures 12, 13 and 14**

8.1.5 Any correction to the workorder that greatly impacts the workorder such as a significant change in holding time or turn around time, the addition of new tests, and/or the addition or cancellation of samples must also be verbally communicated to the appropriate lab supervisor by the project manager. An email is also an effective means of communication.

8.2 REVIEW of the Work Order

8.2.1 The Mitkem Project folders are handed out by the Project Managers. It is the responsibility of the project manager to ensure that the sample ID, date and time of sample collection, date of sample receipt and the correct testcodes/associated preparation testcodes are logged in for each sample. Corrections are made, if necessary, and a new workorder printed out and placed in the red folder.

8.2.2 When adding a workorder that is not going to be invoiced (Mitkem work, PE, etc…) or if the workorder is cancelled one of the following steps must be performed:

1) The workorder client name is changed to Mitkem.
2) All samples are designated as non-billable
3) For PE samples the characters "PE" is added to the SDG number field.
4) If the workorder is to be cancelled with no billable fractions and/or tests, the following steps should be followed:
   - Fill in Log Review with the date.
   - Click on Cancel Work Order button
   - The Completed, Validated, Reported, Faxed, Mailed and Invoiced fields will be populated with the current date. The Project No:, Case No:, SDG No: and EDD By: fields will be populated with Canceled.
5) If the workorder is to be cancelled but some fractions and/or test codes can be billed, then follow these steps:
   - One sample should be logged in with NO-TEST and the correct priced added.
   - Fill in Log Review with the date.
   - Click on Cancel Work Order button
   - The Completed, Validated, Reported, Faxed, Mailed and Invoiced fields will be populated with the current date. The Project No:, Case No:, SDG No: and EDD By: fields will be populated with Canceled.
   - An invoice can be generated.

9 Data Reduction and Calculations

Not applicable

10 Quality Assurance / Quality Control

The Project Manager reviews the workorder within one day of sample receipt. Any changes or corrections are implemented at this step.

11 Data validation and Reporting

Not applicable

12 Corrective Action Procedures

Not applicable

13 Health and Safety

Not applicable

14 Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15 References

Not applicable

Attachments

Figure 1: Main page LIMS
Figure 2: Workorder screen showing Client ID
Figure 3: Project field in workorder
Figure 4: ADD Sample to workorder
Figure 5:AFCEE tab
Figure 6: Add Fraction and Multilog option tabs
Figure 7:POSToption in Multilog
Figure 8: Sample choice box for Matrix Spike
Figure 9: Samples on Hold
Figure 10: Workorder report print tab
Figure 11: Workorder report example
Figure 12: Report tab
Figure 13: Client Report screen, option "base report"
Figure 14: Invoice example

Figure 1: Main page LIMS

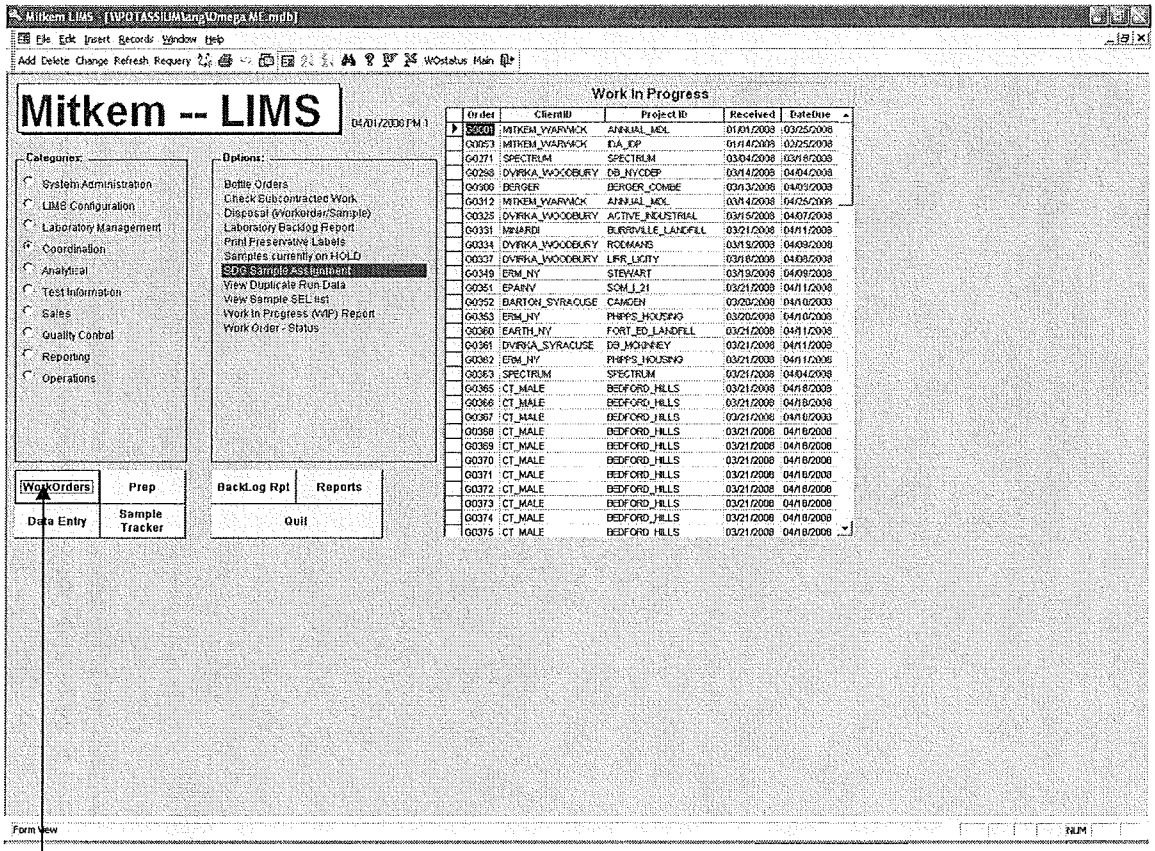
Mitkem LIMS - [\\POTASSIUM\eng\Omega ME.mdb]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
Mitkem -- LIMS
04/01/2008 PM 1
Categories:
System Administration
LIMS Configuration
Laboratory Management
Coordination
Analytical
Test Information
Sales
Quality Control
Reporting
Operations
Options:
Bottle Orders
Check Subcontracted Work
Disposal (Workorder/Sample)
Laboratory Backlog Report
Print Preservative Labels
Samples currently on HOLD
SDG Sample Assignment
View Duplicate Run Data
View Sample SEL list
Work In Progress (WIP) Report
Work Order - Status
WorkOrders
Prep
Data Entry
Sample Tracker
BackLog Rpt
Reports
Quit
Work In Progress
Order | ClientID | Project ID | Received | DateDue
G0001 | MITKEM_WARWICK | ANNUAL_MDL | 01/01/2008 | 03/25/2008
G0053 | MITKEM_WARWICK | IDA_IDP | 01/14/2008 | 02/25/2008
G0271 | SPECTRUM | SPECTRUM | 03/04/2008 | 03/18/2008
G0298 | DVIRKA_WOODBURY | DB_NYCDEP | 03/14/2008 | 04/04/2008
G0300 | BERGER | BERGER_COMBE | 03/13/2008 | 04/09/2008
G0312 | MITKEM_WARWICK | ANNUAL_MDL | 03/14/2008 | 04/25/2008
G0325 | DVIRKA_WOODBURY | ACTIVE_INDUSTRIAL | 03/15/2008 | 04/07/2008
G0331 | MINARDI | BURRVILLE_LANDFILL | 03/21/2008 | 04/11/2008
G0334 | DVIRKA_WOODBURY | RODMANS | 03/18/2008 | 04/08/2008
G0337 | DVIRKA_WOODBURY | LIKR_LICITY | 03/18/2008 | 04/08/2008
G0349 | ERM_NY | STEWART | 03/19/2008 | 04/09/2008
G0351 | EPANY | SOM_I_21 | 03/21/2008 | 04/11/2008
G0352 | BARTON_SYRACUSE | CAMDEN | 03/20/2008 | 04/10/2008
G0353 | ERM_NY | PHIPPS_HOUSING | 03/20/2008 | 04/10/2008
G0360 | EARTH_NY | FORT_ED_LANDFILL | 03/21/2008 | 04/11/2008
G0361 | DVIRKA_SYRACUSE | DB_MOHNNEY | 03/21/2008 | 04/11/2008
G0362 | ERM_NY | PHIPPS_HOUSING | 03/21/2008 | 04/11/2008
G0363 | SPECTRUM | SPECTRUM | 03/21/2008 | 04/04/2008
G0365 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
G0366 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
G0367 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
G0368 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
G0369 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
G0370 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
G0371 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
G0372 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
G0373 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
G0374 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
G0375 | CT_MALE | BEDFORD_HILLS | 03/21/2008 | 04/18/2008
Form View
NUM

Figure 2: Workorder screen showing Client ID

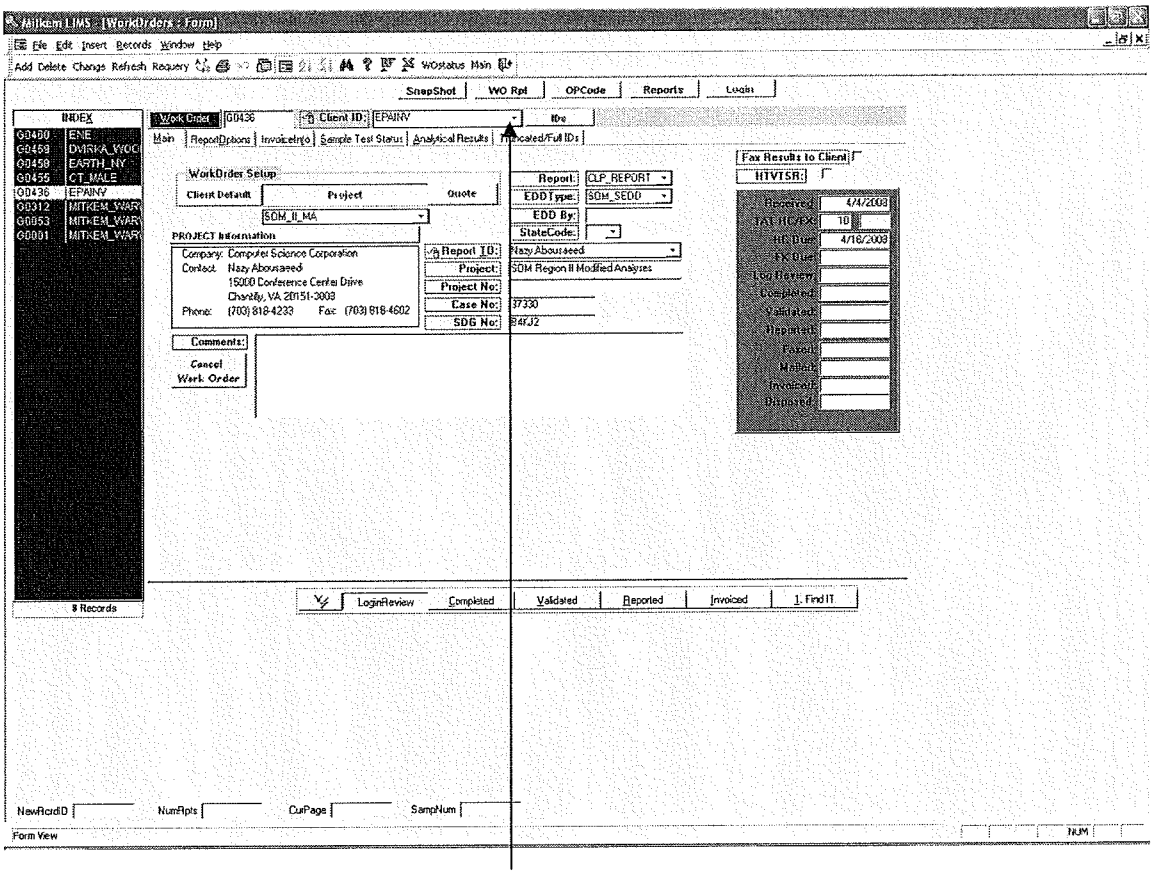
Mitkem LIMS - [WorkOrders - Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
SnapShot
WO Rpt
OPCode
Reports
Login
INDEX
G0400 ENE
G0459 DVIRKA_WOO
G0458 EARTH_NY
G0455 CT_MALE
G0436 EPAINV
G0312 MITKEM_WAR
G0053 MITKEM_WAR
G0001 MITKEM_WAR
8 Records
Work Order
G0436
Client ID:
EPAINV
Main
ReportOptions
InvoiceInfo
Sample Test Status
Analytical Results
WorkOrder Setup
Client Default
Project
Quote
SOM_II_MA
PROJECT Information
Company: Computer Science Corporation
Contact: Nazy Abousaeed
15000 Conference Center Drive
Chantilly, VA 20151-3008
Phone: (703) 818-4233
Fax: (703) 818-4602
Report:
CLP_REPORT
EDD Type:
SOM_SEDD
EDD By:
StateCode:
Report ID:
Nazy Abousaeed
Project:
SOM Region II Modified Analyses
Project No:
Case No:
37330
SDG No:
B4FJ2
Comments:
Cancel Work Order
Fax Results to Client
HTVTSR:
Received
4/4/2008
TAT HC/PX
10
HC Due
4/18/2008
FX Due
Log Review
Completed
Validated
Reported
Faxed
Mailed
Invoiced
Disposed
LoginReview
Completed
Validated
Reported
Invoiced
1. Find IT
NewRecID
NumRpts
CurPage
SampNum
Form View
NUM

Figure 3: Project field in workorder

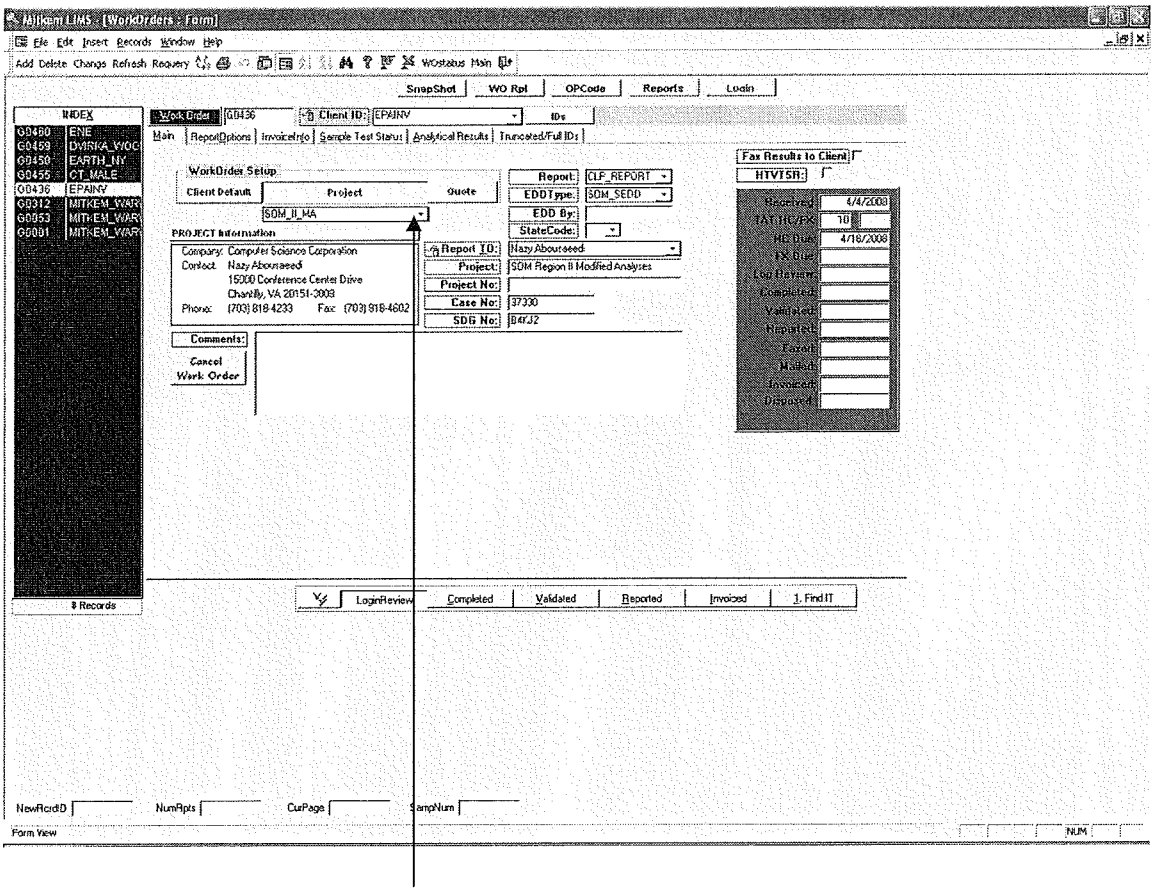
Mitkem LIMS - [WorkOrders - Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
SnapShot
WO Rpt
OPCode
Reports
Login
INDEX
G0460 ENE
G0459 DVIRKA_WOC
G0458 EARTH_NY
G0455 CT_MALE
G0436 EPAINV
G0312 MITKEM_WAR
G0053 MITKEM_WAR
G0001 MITKEM_WAR
8 Records
Work Order
G0436
Client ID:
EPAINV
IDs
Main
ReportOptions
InvoiceInfo
Sample Test Status
Analytical Results
Truncated/Full IDs
WorkOrder Setup
Client Default
Project
Quote
SOM_II_MA
PROJECT Information
Company: Computer Science Corporation
Contact: Nazy Abousseed
15000 Conference Center Drive
Chantilly, VA 20151-2003
Phone: (703) 818-4233
Fax: (703) 818-4602
Report:
CLF_REPORT
EDDType:
SOM_SEDD
EDD By:
StateCode:
Report TO:
Nazy Abousseed
Project:
SOM Region II Modified Analyses
Project No:
Case No:
37330
SDG No:
B4FJ2
Comments:
Cancel Work Order
Fax Results to Client
HTVTSR:
Received
4/4/2008
TAT HC/FX
10
HC Due
4/18/2008
FX Due
Log Review
Completed
Validated
Reported
Faxed
Mailed
Invoiced
Disposed
LoginReview
Completed
Validated
Reported
Invoiced
1. Find IT
NewRecID
NumRpts
CurPage
SampNum
Form View
NUM

Figure 4: ADD Sample to workorder

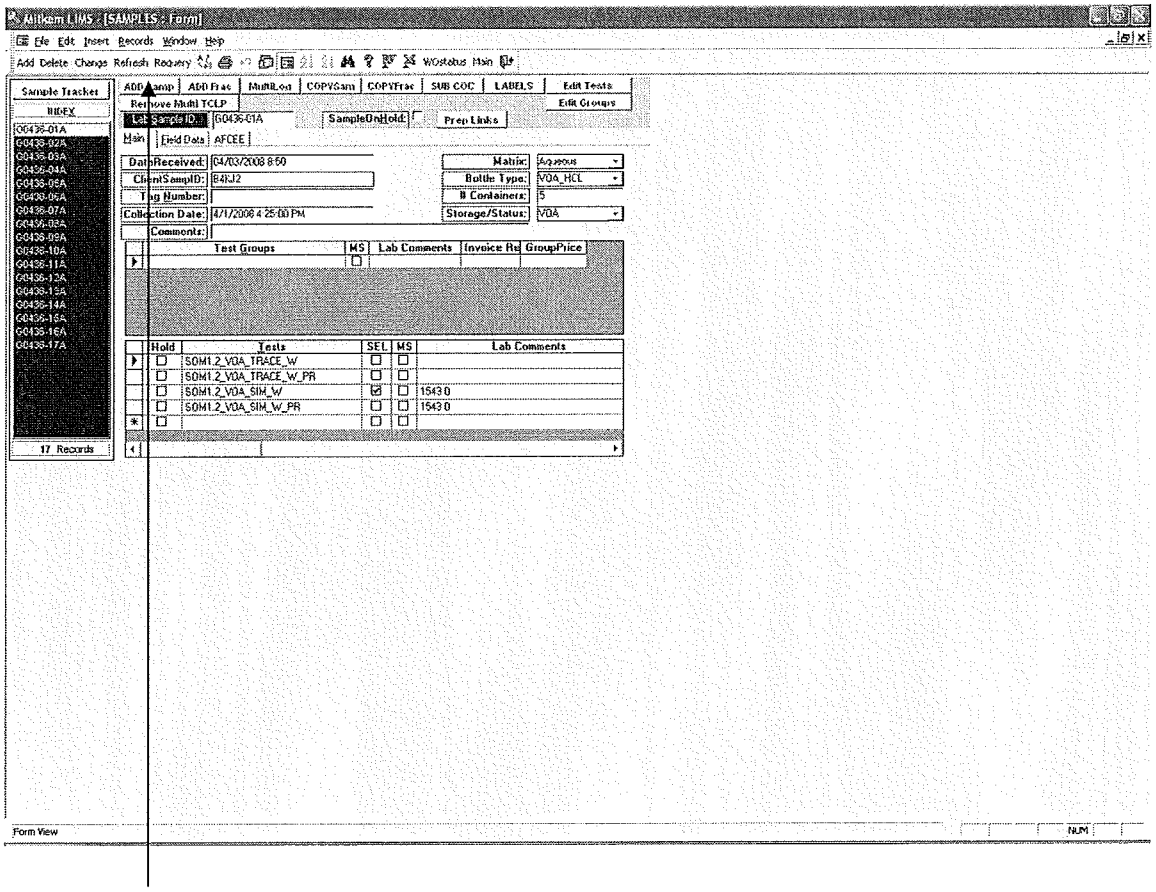
Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Recovery
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLoad
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Remove Multi TCLP
Edit Groups
Lab SampleID:
G0436-01A
SampleOnHold:
Prep Links
Main
Field Data
AFCEE
DateReceived:
04/03/2008 8:50
ClientSamplID:
B4KJ2
Tag Number:
Collection Date:
4/1/2008 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
15430
SOM1.2_VOA_SIM_W_PR
15430
Form View
NUM

Figure 5:AFCEE tab

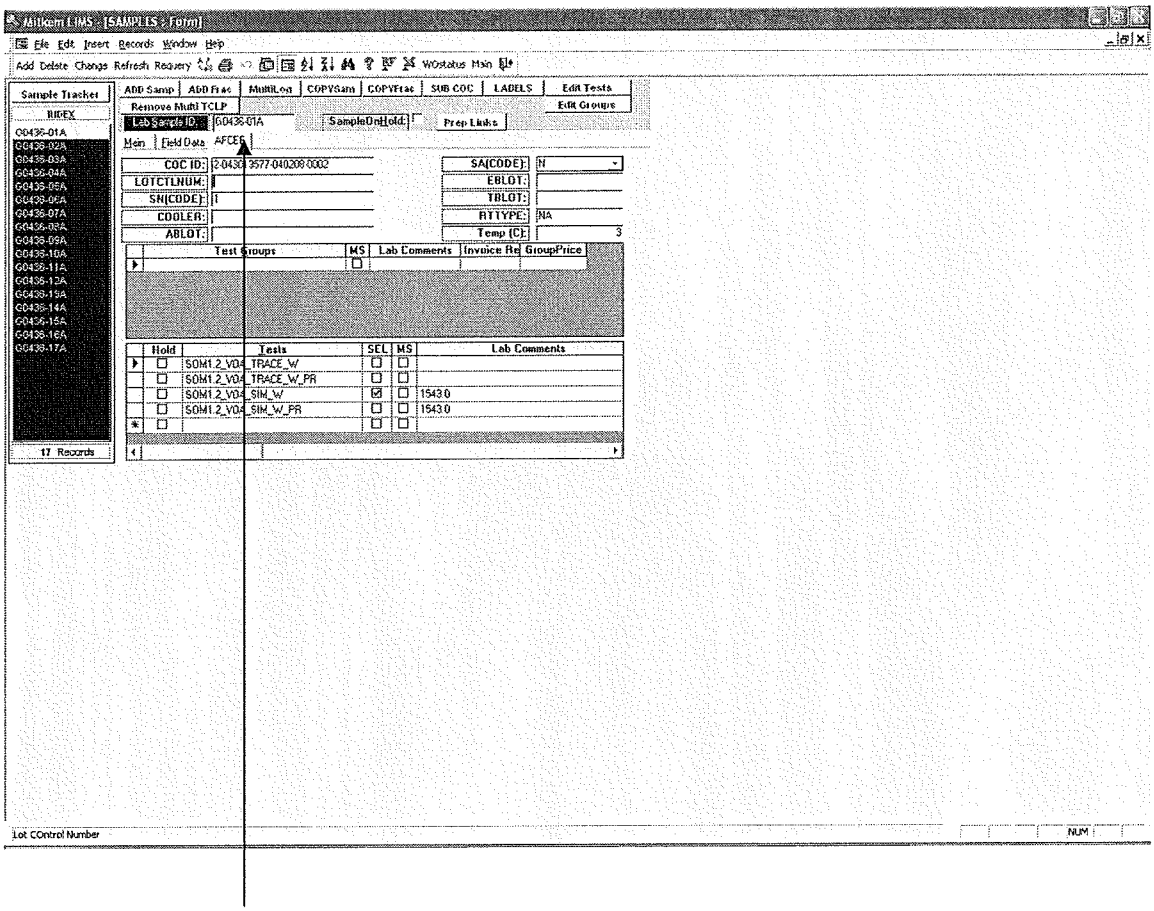
Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Remove Multi TCLP
Edit Groups
Lab Sample ID:
G0436-01A
SampleOnHold:
Prep Links
Main
Field Data
AFCEE
COC ID:
LOTCTLNUM:
SN(CODE):
COOLER:
ABLOT:
SA(CODE):
N
EBLOT:
TBLOT:
RTTYPE:
NA
Temp (C):
3
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_V04_TRACE_W
SOM1.2_V04_TRACE_W_PR
SOM1.2_V04_SIM_W
15430
SOM1.2_V04_SIM_W_PR
15430
Lot COntrol Number
NUM

Figure 6: Add Fraction and Multilog option tabs

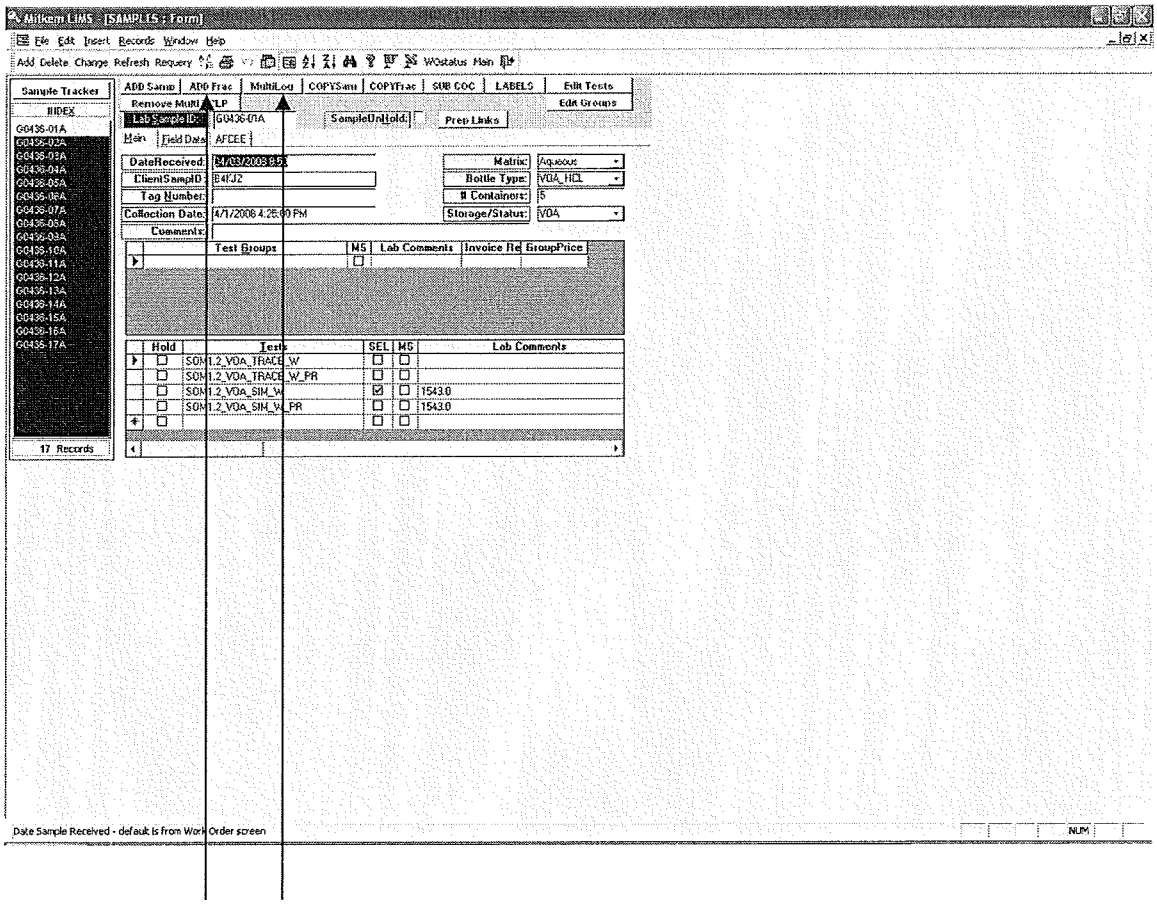
Mitkem LIMS [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSamp
COPYFrac
SUB COC
LABELS
Edit Tests
Remove MultiLP
Edit Groups
Lab Sample ID:
G0436-01A
SampleUnHold:
Prep Links
Main
Field Data
AFCEE
DateReceived:
ClientSampID:
B4FJ2
Tag Number:
Collection Date:
4/1/2006 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
1543.0
SOM1.2_VOA_SIM_W_PR
1543.0
Date Sample Received - default is from Work Order screen
NUM

Figure 7:POSToption in Multilog

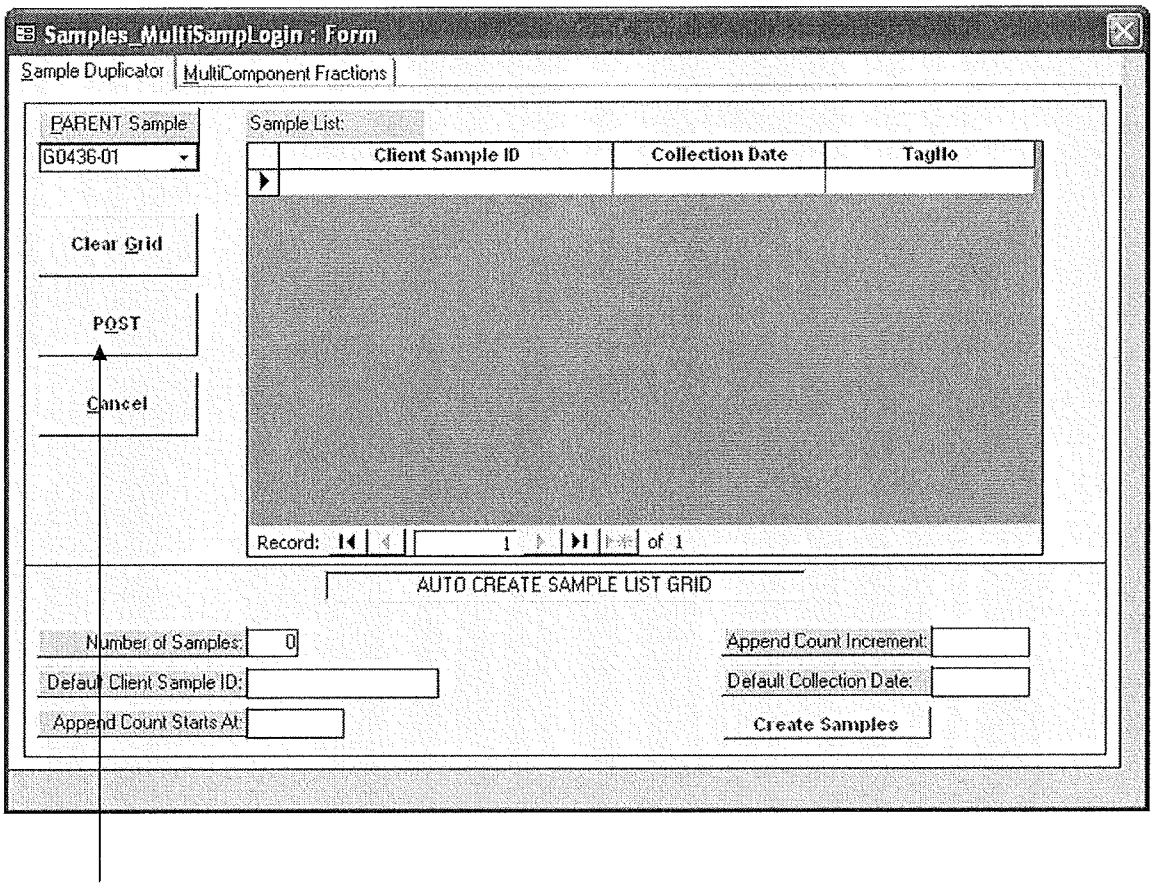
Samples_MultiSampLogin : Form
Sample Duplicator
MultiComponent Fractions
PARENT Sample
G0436-01
Sample List:
Client Sample ID
Collection Date
Taglio
Clear Grid
POST
Cancel
Record:
1
of 1
AUTO CREATE SAMPLE LIST GRID
Number of Samples:
0
Default Client Sample ID:
Append Count Starts At:
Append Count Increment:
Default Collection Date:
Create Samples

Figure 8: Sample choice box for Matrix Spike

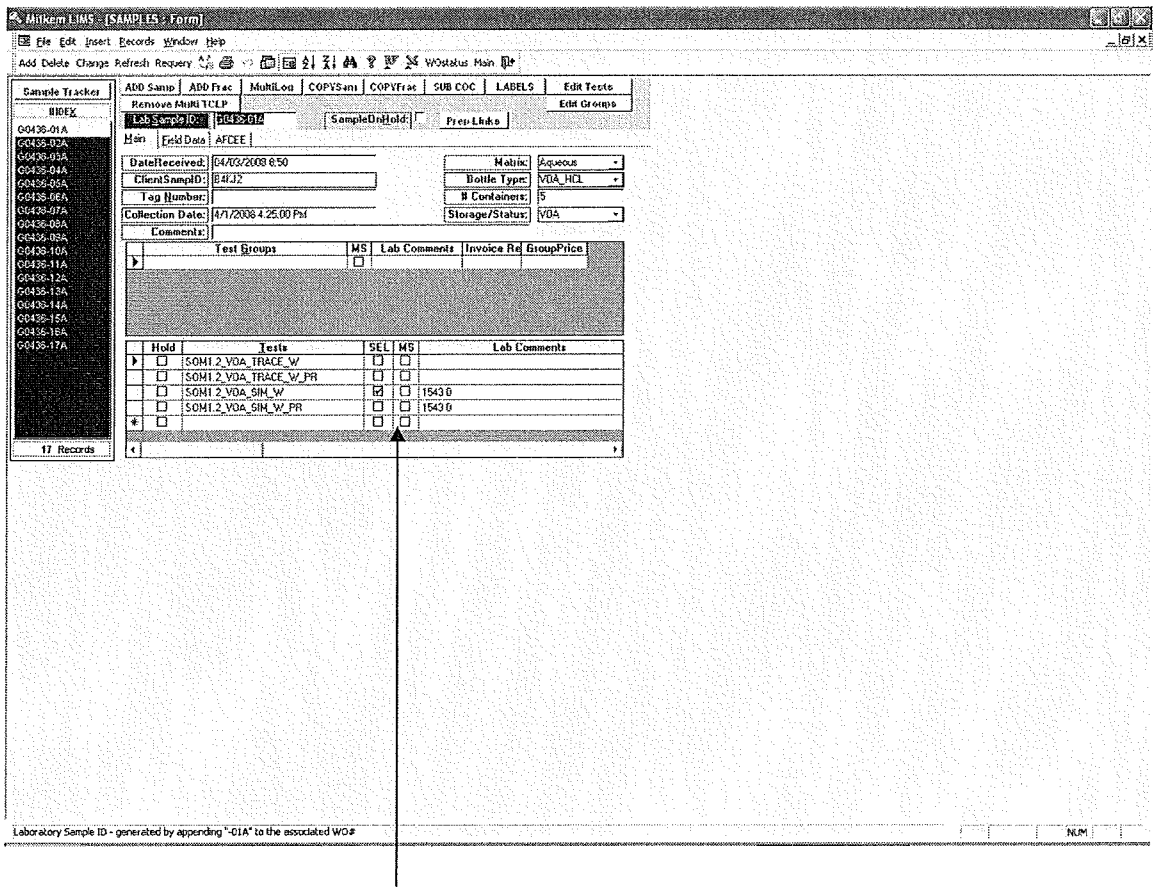
Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery WOstatus Main
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Remove Multi TCLP
Edit Groups
Lab Sample ID:
G0436-01A
SampleOnHold:
Prep-Links
Main
Field Data
AFCEE
DateReceived:
04/03/2008 8:50
ClientSampID:
B4FJ2
Tag Number:
Collection Date:
4/1/2008 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
15430
SOM1.2_VOA_SIM_W_PR
15430
Laboratory Sample ID - generated by appending "-01A" to the associated WO#
NUM

Figure 9: Samples on Hold

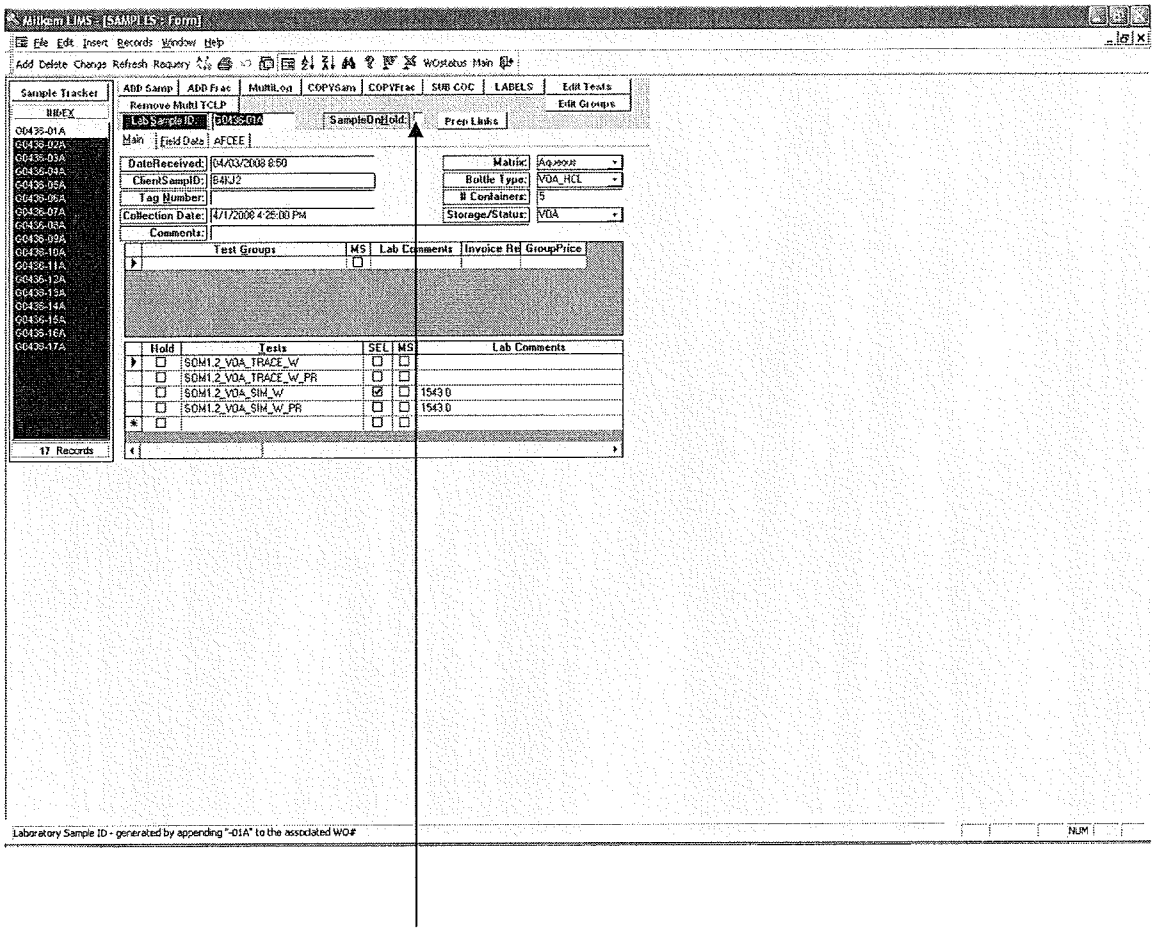
Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Remove Multi TCLP
Edit Groups
Lab Sample ID:
G0436-01A
SampleOnHold:
Prep Links
Main
Field Data
AFCEE
DateReceived:
04/03/2008 8:50
ClientSampID:
B4KJ2
Tag Number:
Collection Date:
4/1/2008 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
15430
SOM1.2_VOA_SIM_W_PR
15430
Laboratory Sample ID - generated by appending "-01A" to the associated WO#
NUM

Figure 10: Workorder report print tab

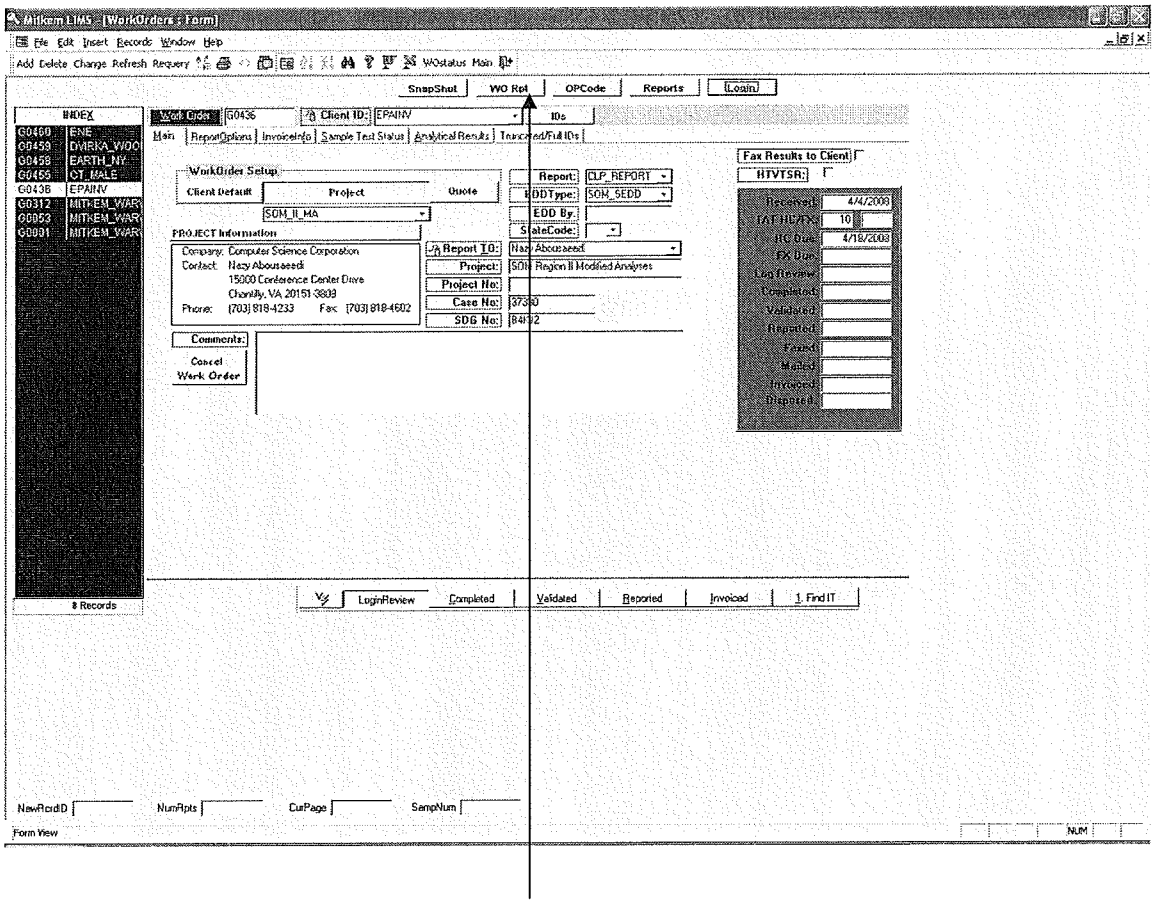
Mitkem LIMS - [WorkOrders : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
SnapShot
WO Rpt
OPCode
Reports
Login
INDEX
G0460 ENE
G0459 DVIRKA_WOO
G0458 EARTH_NY
G0456 CT_MALE
G0436 EPAINV
G0312 MITKEM_WAR
G0053 MITKEM_WAR
G0001 MITKEM_WAR
8 Records
Work Order
G0436
Client ID:
EPAINV
IDs
Main
ReportOptions
InvoiceInfo
Sample Test Status
Analytical Results
Transcend/Full IDs
WorkOrder Setup
Client Default
Project
Quote
SOM_II_MA
PROJECT Information
Company: Computer Science Corporation
Contact: Nazy Abousseedi
15000 Centerence Center Drive
Chantilly, VA 20151-3809
Phone: (703) 818-4233 Fax: (703) 818-4602
Report:
CLP_REPORT
EDDType:
SOM_SEDD
EDD By:
StateCode:
Report TO:
Project:
Project No:
Case No:
37380
SDG No:
Comments:
Cancel Work Order
Fax Results to Client
HTVTSR:
Received
4/4/2008
TAT HL7Rcd
10
HC Due
4/18/2008
FX Due
Log Review
Completed
Validated
Reported
Found
Mailed
Invoiced
Disputed
LoginReview
Completed
Validated
Reported
Invoiced
1. FindIT
NewRecID
NumRpts
CurPage
SampNum
Form View
NUM

Figure 11: Workorder report example

Mitkem LIMS - [WDSummary - Report]

File Window Help

Add Delete Change Refresh Requery WOstatus Main

100% Close

Mitkem Laboratories *08/Apr/08 16:19* **WorkOrder: G0436**

Client ID: EPAINV
Project: SOM Region II Modified Analyses
Location:
Comments: N/A

Case: 37330
SDG: B4KJ2
PO: EP-W-05-030

Report Level: CLP_REPORT
EDD: SOM_SEDD
HC Due: 04/18/08
Fax Due:

Sample ID	HS Client Sample ID	Collection Date	Date Rec'd	Matrix	Test Code	Lab Test Comments	Bold	MS	SEL	Storage
G0436-01A	B4KJ2	04/01/2008 16:25	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-02A	B4KJ3	04/01/2008 12:20	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-03A	B4KJ4	04/01/2008 16:20	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-04A	B4KJ5	04/01/2008 17:30	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-05A	B4KJ7	04/01/2008 9:30	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-06A	B4KK0	04/01/2008 8:00	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-07A	B4KH4	04/03/2008 11:10	04/04/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA

Client Rep: Agnes R Ng

Page 1 of 3

Page: 1

Ready NUM

Figure 12: Report tab

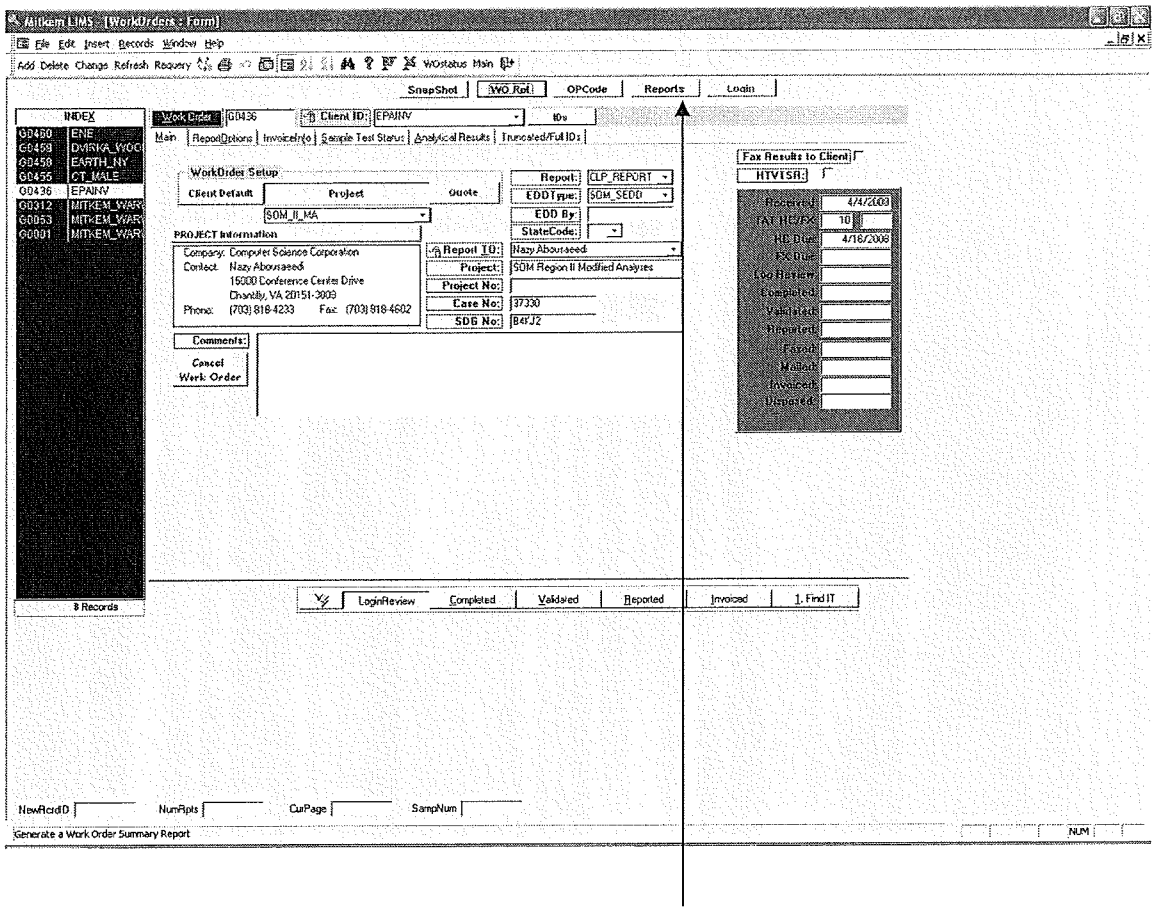
Mitkem LIMS - [WorkOrders : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
SnapShot
WO Rel
OPCode
Reports
Login
INDEX
GD460 ENE
GD459 DMRKA_WOO
GD458 EARTH_NY
GD455 CT_MALE
GD436 EPAINV
GD312 MITKEM_WAR
GD053 MITKEM_WAR
GD001 MITKEM_WAR
8 Records
Work Order
GD436
Client ID:
EPAINV
Main
ReportOptions
InvoiceInfo
Sample Test Status
Analytical Results
Truncated/Full ID:
WorkOrder Setup
Client Default
Project
Quote
SOM_II_MA
Report:
CLP_REPORT
EDDType:
SOM_SEDD
EDD By
StateCode:
PROJECT Information
Company: Computer Science Corporation
Contact: Nazy Aboutaseed
15000 Conference Centre Drive
Chantilly, VA 20151-3009
Phone: (703) 818-4233 Fax: (703) 818-4602
Report TO:
Nazy Aboutaseed
Project:
SOM Region II Modified Analyses
Project No:
Care No:
97330
SDG No:
B4FJ2
Comments:
Cancel Work Order
Fax Results to Client
HTVISR:
Received:
4/4/2003
TAT HC/PX
10
HC Due:
4/16/2003
PX Due:
Lab Review:
Completed:
Validated:
Reported:
Faxed:
Mailed:
Invoiced:
Deported:
LoginReview
Completed
Validated
Reported
Invoiced
1. Find IT
NewRecID
NumRpts
CurPage
SampNum
Generate a Work Order Summary Report
NUM

Figure 13: Client Report screen, option "base report"

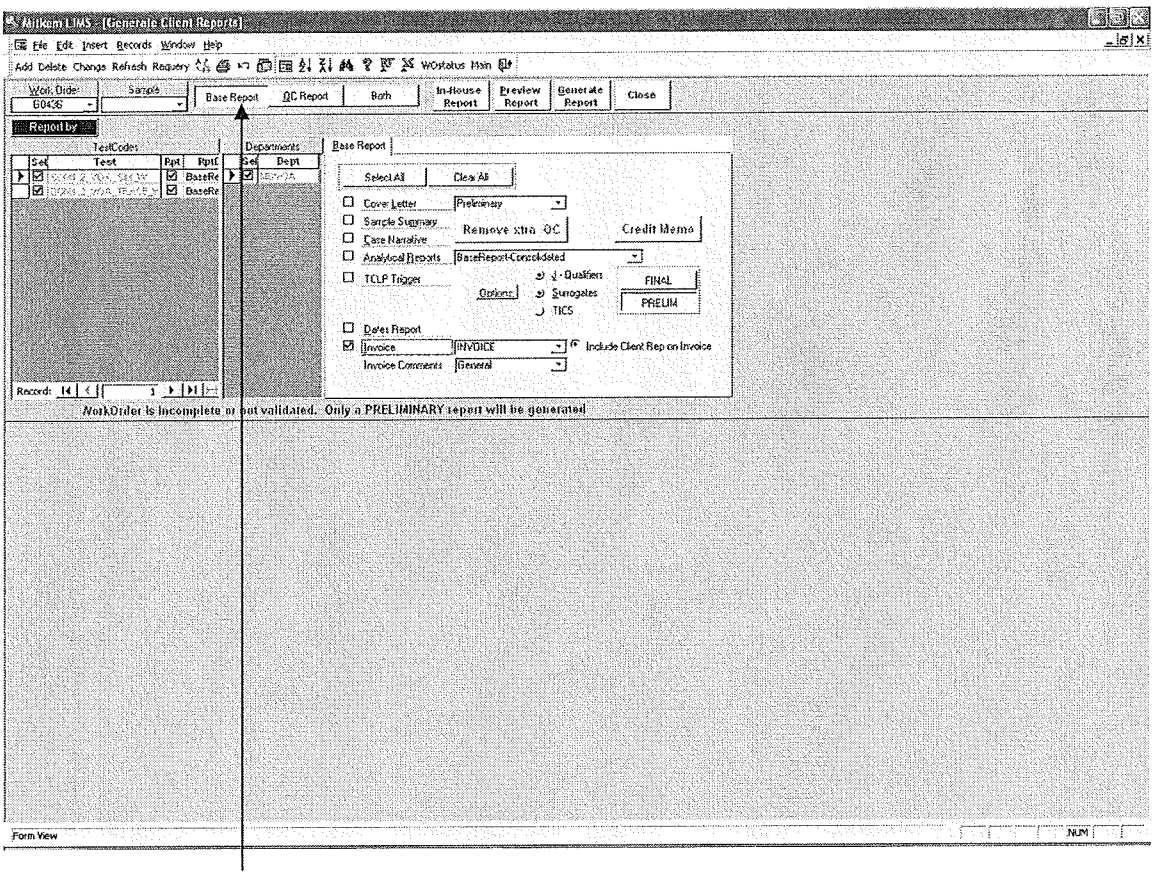
Mitkem LIMS - [Generate Client Reports]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOStatus Main
Work Order
Sample
Base Report
QC Report
Both
In-House Report
Preview Report
Generate Report
Close
Report by
TestCodes
Departments
Base Report
Select All
Clear All
Cover Letter
Preliminary
Sample Summary
Remove xtra QC
Credit Memo
Case Narrative
Analytical Reports
BaseReport-Consolidated
TCLP Trigger
Qualifiers
Surrogates
TICS
Options
FINAL
PRELIM
Delet Report
Invoice
INVOICE
Include Client Rep on Invoice
Invoice Comments
General
Record:
WorkOrder is incomplete or not validated. Only a PRELIMINARY report will be generated
Form View
NUM

Figure 14: Invoice example

Mitkem LIMS - [Invoice__rpt : Report]

Bill To:

INVOICE

EPA Financial Management Center
Attn:
Research Triangle Park
Research Triangle Park, NC 27711

Number:
**G0436
Date:

Client Rep: Nazy Abousaeedi
Work Order: G0436
PO Number: EP-W-05-030
Date Received: 04/04/2008

Project No:
Project Name: SOM Region II Modified Analyses
Case No: 37330
SDG No: B4KJ2

Test	Remarks	Matrix	Qty	Price	Test Total
SOM1.2_VOA_SIM_W	1543.0	Aqueous	17	$1.00	$17.00
SOM1.2_VOA_TRACE_W		Aqueous	17	$85.00	$1,445.00

Subtotal:	$1,462.00
Discount:	0.00%
Surcharge:	0.00%
Misc Charges:	$0.00
Payment Received:	$0.00
INVOICE Total:	$1,462.00

All invoices are due and payable net 30 days from receipt.

Page: 1

Ready

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Receipt, Storage, Tracking and Disposal

SOP No. 30.0003

Rev. 14

	Signature	Date
QA Director:		2/3/10
Lab Director:		2/4/10
Effective Date:	2/11/10	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Receipt, Storage, Tracking and Disposal

Rev. 14

1. Scope and Application

This Standard Operating Procedure describes the procedures that must be followed upon receipt of samples at MITKEM. It describes the procedures to log-in and store samples. It also describes procedures for checking samples out of the storage area for analysis, and for final disposal of samples. Detailed procedures for entering work orders and samples into Mitkem's Laboratory Information Management System (LIMS) are described in a separate SOP Number 20.0003.

2. Personnel Qualifications and Responsibilities

The Sample Custodian should have a two-year degree in environmental science or a related field or have six months of on-the-job experience working with a trained and qualified Sample Custodian.

3. Summary of Procedure

When sample coolers are received, cooler custody seals and temperature are checked. The chain-of-custody forms are signed and the condition of the samples are checked, including any sample custody seals. Each sample is assigned a specific individual sample number. Any discrepancies are documented and resolved. Samples are logged-in to the LIMS following procedures in SOP 20.0003. Labels are generated, and affixed to samples. The pH of aqueous samples (Inorganic only, never VOA) are checked and if need be, either acid or base is added. The addition of preservative (and its lot number) is recorded on the Condition Form. Samples are stored in secure area under documented custody conditions. Certain types of VOC soil samples require special handling, as described in **Section 8.2.2**. Samples are kept at Mitkem for a specific time as called for by contract with the client, and then disposed of in accordance with federal, state and local regulations. The door to the sample receiving room is locked at all times, using a keypad code lock.

4. Sample Preservation, Containers, Handling, and Storage

Samples received at Mitkem are collected by our clients. Mitkem does provide preservatives and sample containers pre-cleaned by bottle suppliers.

Tables 7-1, 7-2 and 7-3 of Mitkem's Quality Assurance Plan (QAP) lists the type of container needed for a particular type of analyte and the proper preservative.

5. Interferences and Potential Problems

Any problem with sample condition including custody seals, breakage, discrepancies in chain of custody or other shipping documentation, cooler temperature, missing samples, etc. must be communicated to the client promptly and resolved. The Sample Custodian or assistant documents the problem and notifies the Mitkem Project Manager, who will then notify the client. For CLP the Project Manager will notify the Sample Management Office (SMO).

6. Equipment and Apparatus

6.1 NIST-calibrated temperature "gun".

6.2 Condition Forms: Non-CLP, CLP (DC-1).

6.3 Orange colored Sample Condition Notification forms.

6.4 Narrow range pH paper, capable of recording pH 0- 2.5, and 11-13.

6.5 Computer with Accessories to run the LIMS system.

6.6 Label printer and labels.

7. Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test.

7.1 Hydrochloric Acid (HCl), Trace Metal Grade.

7.2 Nitric Acid (HNO_3), Trace Metal Grade.

7.3 Sodium Hydroxide (NaOH), Certified A.C.S.

7.4 Zinc Acetate $(CH_3CO_2)_2$ Zn $2H_2O$, certified.

7.5 Sulfuric Acid (H_2SO_4), Trace Metal Grade.

7.6 Phosphoric Acid (H_3PO_4), Trace Metal Grade.

Reagents are ordered by the inorganic laboratory section, and kept in the sample receiving and bottle prep areas. Zinc Acetate is stored in the volatile laboratory as it is only used with volatile sample bottle prep.

If the reagent needed is in neat form (NaOH or Zinc Acetate) a new bottle may be obtained from the chemical storage area. If you take the next to the last or the last bottle for login dept use, you must inform the inorganic lab supervisor so more can be ordered.

8. Procedure

8.1 Log-In Procedures:

Samples received at Mitkem are received from private courier (FedEx, UPS, etc.) or Mitkem courier or delivery by the clients. All samples received must be accompanied by a chain-of-custody (COC) record.

8.1.1 Prior to opening coolers the receiving hood must be turned on, and safety glasses, lab coat, and gloves worn.

8.1.2 Select the next sequential red workorder folder with a pre-assigned work order number. Work order numbers consist of one alpha character followed by a four digit number YXXXX, in which Y is the alpha character indicating the number of years that the Omega LIMS System has been operational and XXXX is a sequential number for this workorder for the current year. For example, 2006 is assigned the alpha character "E" and the 5th set of samples received during 2006 is assigned work order number E0005. Occasionally, a client wishes for a workorder number to be ongoing for several shipments of samples (i.e. EPA). The project manager will notify the sample custodian if additional samples are to be added to an existing workorder (an open Sample Delivery Group or SDG).

8.1.3 Before opening coolers, check the outside of the opening of the shipping cooler for the presence of custody seals. Remove any shipping air bills and note the work order number on them. If an EPA cooler is received without an air bill attached, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information. This information should be noted on the Sample Condition Form (See **Attachments 1 and 2**).

NOTE: There are two different types of Sample Condition Forms; one for commercial samples, and one for the US EPA contract samples (see Attachments). Place air bills and all other paperwork for this shipment in the appropriate red workorder folder.

8.1.4 Open the cooler underneath the hood and remove the chain-of-custody form. This is typically located in a plastic zip-lock bag on the top of the samples or taped to the inside of the cooler lid. Write the Mitkem workorder number on the COC. Sign the COC, noting the date and time of receipt of the samples at Mitkem. Look for return air bills to return coolers to the client; these are typically included in EPA projects. If the COC is missing, notify the Mitkem project manager using the Mitkem Sample Condition Notification (printed on orange paper) form immediately. Do not process the samples. Store the samples in the walk-in cooler until the client is contacted and responds.

8.1.5 Take the temperature of the contents using the temperature gun or thermometer.

8.1.5.1 Use the Temperature Gun as follows:

8.1.5.1.1 Unwrap a sample in the opened cooler. Unwrap enough of the sample in order to take a temperature reading.

8.1.5.1.2 Take the temperature by holding the gun no more than two inches from the sample container. Do not aim at the label or cap of the sample. Take several readings on at least 2 samples at opposite ends of the cooler. Record the temperature on the Sample Condition Form and the lower right hand corner of the chain-of-custody or where noted on the Chain-of-Custody. The acceptable temperature range is 4° ± 2°C (2° to 6°C). If the temperature is acceptable, move on to **Section 8.1.6**. If the temperature is not within this range, enter the temperature on the Sample Condition Form, circle "yes" to the question regarding the Sample Notification form, and fill out the Sample Notification form (See **Attachment 3**). For CLP projects, if the temperature exceeds 10°C, the project manager will contact SMO for corrective action guidance. Give the entire folder and the Sample Notification form to the project manager. The project manager will contact the client for instructions on how to proceed and return the folder to the sample custodian indicating action to be taken. During the interim, the samples in question will not be processed, but the closed cooler will be stored in the walk-in cooler.

8.1.5.1.3 Record the Temperature Gun ID on the sample condition form.

8.1.5.2 If a temperature blank is present, use a thermometer as follows:

8.1.5.2.1 Insert the thermometer in the temperature blank.

8.1.5.2.2 Let stand for a minimum of three minutes to as long as five minutes maximum.

8.1.5.2.3 Read the temperature and record it on the Sample Condition Form and in the lower right hand corner of the COC or where noted on the Chain-of-Custody. If the temperature is not within 4° ± 2°C (2° to 6°C), proceed as in **section 8.1.4** above. For CLP projects, if the temperature exceeds 10°C, the project manager will contact SMO for corrective action.

8.1.5.3 A calibration check of the thermometer is to be done quarterly or as the thermometer is replaced by comparing it's reading to the NIST thermometer and recording the results in the QA/QC Thermometer Calibration Logbook. See **SOP No. 110.0006** for the calibration procedure.

8.1.5.4 The presence/absence of a temperature blank in the shipment is documented on the Form DC-1. Follow the instructions on the DC-1 Form for contacting SMO, and for documentation in the event a temperature blank is not present.

8.1.6 If the Sample Custodian is not present, the person receiving the samples should perform all steps through **Section 8.1.5.2.3** then store the cooler in the walk-in refrigerator, R1, until the Sample Custodian returns, after removing any VOC samples. Samples for VOC analysis require special handling, as described in **Section 8.2.2**. In general, if not logged-in immediately, VOA vials must be removed from the cooler and stored in refrigerator R2 until logged-in to the LIMS. If VOC samples are received in Encore devices (typically contained in silver foil pouches), or are noted as requiring freezing to preserve, they must be removed and stored in the freezer F2. Encore samples must be extruded into pre-weighed VOC vials as soon as possible. Contact one of the following to insure this occurs: VOA Laboratory Supervisor, Sample Custodian, Project Manager, Laboratory Director or QA Director. Leave the paperwork in the red folder on the sample custodian's desk.

8.1.7 Once all of the coolers received have been inspected as above, prioritize them for logging into the LIMS with fastest turn-around times and short holding times processed first. The labs should be notified immediately about quick-turn analyses. This can be done either verbally, or by copying the COC and distributing that to the laboratories.

8.1.8 Remove and inspect samples from the coolers for the first workorder. Sample containers are removed from the cooler and lined-up on the counter in the same order as listed on the chain of custody form. Check to see that the correct number of samples is present. Check to see if any samples are broken. Check aqueous VOA sample vials for the presence of air bubbles or headspace. If bubbles or headspace are present, a Sample Notification form should be filled out and given to the Project Manager. Check to see that the quantity present is sufficient for the test requested. Make sure the client IDs on the bottles agree with the COC. Check the custody seals, if any, on the sample containers to ensure the all seals are present and intact.

8.1.9 For U.S. EPA samples, sign the COC and note the temperature on the bottom right corner of the COC. Place the project number in the top right hand corner of the COC. Record the following information on the Form DC-1.

- Sign DC-1. Note that the person signing should be the person who signed for receipt on the COC. There will be one DC-1 form for each cooler received for a particular case. Only the samples in that particular cooler will be listed on that DC-1 form.
- EPA Case number from the COC.
- List sample tag control numbers and compare these with the COC records.
- Document whether or not these numbers agree.
- If Sample tag numbers are not listed on the COC form, record this fact.
- Record the custody seal numbers if present.
- Write the work order number on the air bill and place it in the red project folder. Record the name of the courier and the air bill number on Form DC-1.
- Date of receipt.
- Time of receipt.
- EPA sample numbers.
- SDG-Final Sample must be documented on EPA TR/COC.
- SDG (Sample Delivery Group) number. The SDG is the lowest alphanumeric sample on the first shipment and remains the same until the work order is closed.
- EPA sample numbers.
- Assigned Lab ID numbers. Note that with EPA projects, shipments may be received over several days to be added to the same work order, up to twenty field samples, not including PE samples. The SDG number stays the same, but the sample ID numbers run consecutively.
- Whether samples were delivered by hand or not.
- Any problems and discrepancies in "remarks" box.
- The cooler temperature is entered in item #10 of the Form DC-1.

Note from the COC whether the work order is closed, or if more samples are expected to be added (work order/SDG is still open). EPA projects are closed

automatically after one week from the date of receipt, for cases with a turnaround time of 14 or 21 days and three days for samples with a turnaround time of 7 days, of the first shipment or sooner if the client checks the box that the project is closed.

Discrepancies in EPA shipments should be reported to the Project Manager directly. Any irregularities in the above information should be documented and the client contacted by either Project Manager or the Sample Custodian. Until an answer is received from the client, samples should be stored in the walk-in cooler.

If there are any discrepancies, note them on the Sample Condition Notification Form and give the form and the workorder folder to the project manager. Samples with issues will not be processed further, but stored in the walk-in cooler until a resolution has been reached with the client. For CLP (EPA) samples, the Sample Management Office (SMO) will be contacted for corrective action. Communication of the corrective action will be placed in the red project folder.

8.1.10 Log the workorder and samples into the Laboratory Information Management System (LIMS) following procedures described in SOP 20.0003. Labels are generated and attached to each sample container. In the case of pre-preserved samples in VOA vials (sodium bisulfate, DI water and/or methanol), be sure the Mitkem sample ID label does not cover the tare weight of the vials.

8.1.11 Have the project manager or a second person check the Lab IDs on the labels vs. the client IDs on the COC to confirm the bottles have been properly labeled. Ensure the reviewer initials the proper line of the Sample Condition Form or DC-1 form.

8.1.12 Enter the workorder number, date received, and client name in the Sample Receipt and Tracking Logbook (**Attachment 6**). Occasionally, a client wishes for a project number to be ongoing for several shipments of samples (i.e. EPA). In such cases the date of arrival of each subsequent shipment should be noted for the original work order number.

8.1.13 Inorganic preserved samples must now be tested for pH as follows:

For metals, wet chem, etc., pour a small amount of sample over a strip of pH paper that is suspended over the mouth of a waste container. The pH of the acidified samples is taken by using the narrow range pH strips which measures pH 0 to 2.5. The sample pH should be less than two, as indicated by the color on the pH indicator package. The pH of the basic samples is taken by using the narrow range pH strips which measures pH 11.0 – 13.0. The basic samples should have a pH 12 or greater, as indicated by the color of the pH indicator package. (See **Attachment 4** for instructions on the use of the pH paper.) Acidified samples must have a pH of less than 2. Any pH adjustments that need to be made are recorded on the Sample Condition Form for the workorder.

Record the lot number and ID of the reagent used and the approximate volume used to adjust the pH. For metals samples requiring acidification, alert the inorganic laboratory to allow 24 hours before metals preparation to allow enough time for the acids to dissolve any metals that adsorb to container walls.

8.1.14 Some samples (i.e. fecal coliform bacteria) which require subcontracting must be done so immediately as the analysis has an extremely short hold time. A list of analyses typically subcontracted can be printed from the LIMS Omega system. An Outside Services Purchase Order is also generated from the LIMS system when the batch of samples to subcontract is created.

8.1.15 Receiving personnel make one copy of the sub contract COC. This COC accompanies the samples and is signed by the laboratory that will perform the analysis. A copy of the COC is placed into the red workorder folder and one copy is sent to Accounting.

8.2 Storage and Location of Samples:

8.2.1 Semivolatile Organic, Pesticides/PCBs, and Wet Chemistry Samples:

Samples are stored in the main walk-in refrigerator (R1) in the sample receipt room at 4° ± 2°C. Once the samples have been placed on a shelf, or shelves, record the location(s) in the Sample Receipt and Tracking Logbook. The person putting the samples into the walk-in must put their initials in the logbook beside the location entries.

8.2.2 Volatile Organic Samples:

Samples for VOA are stored separately from all other samples, in refrigerators or freezers dedicated for this purpose. These refrigerators and freezers are located in the VOA Laboratory. A soil sample for VOA may contain one or more of the sample types listed below. Care must be taken to insure each sample vial type receives the proper handling. Soil samples for volatiles analysis must have special handling performed by the VOA lab staff.

A storage blank must accompany all VOA samples requiring EPA/CLP or New York State ASP analyses when placed in the VOA refrigerators. The storage blanks are prepared in the volatiles department using the VOA Type I water. The 40ml vial is filled so there is **no** headspace, capped immediately and tightly, and labeled with the date prepared and any preservative added. The storage blanks for unpreserved low/medium soil samples are prepared by placing approximately 5 g of inert sand in dry closed-system purge-and-trap vials. These inert sand storage blanks will be stored in the same freezer with unpreserved low/medium soil samples.

8.2.2.1 Samples suspected of containing high concentrations of volatile organics must be stored in a separate VOA refrigerator designated for such samples. This isolates these high concentration samples from other samples, and helps to control cross-contamination.

8.2.2.2 Soil samples in pre-weighed, dry closed-system vials (no liquid inside the vial) should be stored in a VOA freezer (containing no standards or sample extracts). These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.3 Soil samples that contain up to 5 mL of water. These vials are also to be stored in a VOA freezer. These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.4 Soil samples that contain 5mL of water containing a sodium bisulfate preservative. These vials are also to be held in a VOA refrigerator (specifically for EPA CLP samples). Samples for Method 8260 analysis may be held in a VOA refrigerator. These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.5 Soil samples received in the Encores (typically contained in silver foil pouches), the samples must be extruded into tared, dry closed-system purge-and-trap vials by the VOA lab staff, and then re-weighed to obtain the final sample weight. This should happen immediately (for EPA CLP samples), but no longer than 48hours of sample collection. The vials are then to be placed into a VOA freezer.

8.2.2.6 Soil samples may also arrive as methanol preserved samples. These samples do not require freezing, but they shall be held in a VOA refrigerator to be weighed by VOA lab staff prior to analysis. If possible, check that the methanol covers the soil samples within the vial. If the methanol is insufficient, note this on the Sample Condition Form and relay the information to the VOA lab.

8.2.2.7 Unpreserved VOA soil samples should be stored in a VOA freezer (EPA CLP samples) or a VOA refrigerator (for Method 8260 samples) until the time of analysis.

8.2.2.8 Water samples are stored in VOA refrigerators. Trace Volatile samples for the EPA/CLP SOM01.2 contract must be stored in a separate refrigerator from all other samples. At present, this has been designated as **R13 and R4**.

8.2.2.9 Log the samples into the VOA lab. Enter the workorder and other pertinent information in the Volatile Receiving Logbook (See

Attachment 7) and sign off that the samples have been relinquished. If the samples are ever removed from VOA they must be signed out of the VOA lab as well.

8.2.3 Metals Samples:

Aqueous preserved samples for metals analyses are stored on the rack in the sample receipt area.

8.2.3.1 ISM water and soil samples require refrigeration at $4^{o} \pm 2^{o}C$ and must be stored in the walk-in. Cyanide aqueous samples must also be protected from light. Only aqueous cyanide samples are required to remain stored in refrigeration after distillation. All other matrices and ISM test methods may be stored at room temperature after preparation, until disposal. Once the samples have been placed on a shelf, or shelves, record the location(s) in the Sample Receipt and Tracking Logbook. The person putting the samples onto the rack must put their initials in the logbook beside the location entries.

Standards must be stored in separate freezers or refrigerators isolated from samples and sample extracts.

8.3 Storage and Location of Extracts:

Extracts for semivolatile analyses must be stored, shielded from light, at $4^{o} \pm 2^{o}C$ in a separate refrigerator stored either in the laboratory or in Unit 3. Extracts for Pest/PCB are stored in refrigerators in the same manner.

Extracts must be stored separately from standards.

Extracts for semivolatile and Pest/PCB must be stored until the proper time to dispose of them per client approval. Mitkem normally retains sample extracts for a period of three months from the date of delivery of the final report to the client

USEPA CLP and New York State ASP extracts must be stored for 365 days after delivery of a complete data package to the client.

8.4 Temperature Monitoring:

The temperature of the storage areas must be monitored on a daily basis, including weekends and holidays. This temperature is recorded electronically using temperature probes that are affixed inside all refrigerator and freezer units. Specific employees are notified of temperatures outside of the acceptance ranges by email so that corrective action can be initiated. If any refrigerator/freezer fails, first investigate the problem. Was the door ajar? Is something preventing the door from shutting fully? If the situation

can be rectified, do so and check the temperature again after a sufficient period of time. Adjust the temperature control knob in the unit if other attempts have not fixed the problem and continue to monitor the temperature. All adjustments and resolutions must be entered into the Temperature Probe Program "Event" log. When the temperature meets criteria, document that the refrigerator/freezer has been "*returned to control*". If there is no obvious issue with the refrigerator/freezer, or the refrigerator does not maintain the correct temperature, the QA Director or Lab Director must be notified immediately to correct the problem. Fill out a report in the LIMS Omega Corrective Action Report. Acceptable temperature ranges are 2° – 6°C for refrigerators and –10°C to -20°C for freezers. The temperatures are recorded electronically multiple times daily. See Mitkem SOP 80.0020, Temperature Monitoring Systems.

8.5 Tracking of Samples and Extracts:

8.5.1 Sample Log-out:

When samples are taken from their storage areas for extraction, they must be signed out using the appropriate Sample Receipt Logbook. During 2010, Mitkem will be transitioning from the existing system to the use of bar-coded scanners to document chain of custody of samples from the storage area into the laboratories for preparation and/or analysis. Presently, the log-out procedure is a two-person procedure that requires samples to be relinquished by either the Sample Custodian or Authorized Laboratory Personnel

The analyst retrieves the samples from the walk-in cooler and the Custodian or Authorized Laboratory Personnel checks the sample numbers. The Mitkem sample numbers as well as the bottle # are recorded (i.e. E0140-02B#2). Both people initial and date the Receiving Logbook in the appropriate places. When the sample is logged-out by an analyst, it is considered to be in the custody of that analyst until he/she returns the sample to its storage area.

Upon the sample's return to the storage area the samples are signed into Receiving using the same procedure. This time the analyst relinquishes the samples and the Custodian or Authorized Laboratory Personnel checks the sample numbers to be returned. Each person signs and dates in the appropriate places in the Receiving Logbook.

Authorized laboratory personnel are employees who are knowledgeable in regard to the receipt and log-in of samples. A list of Authorized Laboratory Personnel is posted on the Receiving bulletin board. (See **Attachment 5**).

8.5.2 Sample Extraction:

If a sample is signed out for extraction, the sample is logged into the lab by entering the Lab ID into the appropriate extraction logbook. After extraction,

any remaining sample volume is returned to the storage area and signed back into the Receiving area for storage as noted in **Section 8.5.1.1**.

When working with samples in the preparation laboratories, the following procedures must be adhered to:

° All activities performed on the sample must be recorded.
° Entries in the logbooks must be signed and dated (mm/dd/yyyy).
° All entries must be made in ink.
° Corrections and additions must be made by drawing a single line through the errors, entering the correct information, and initializing and dating the new information.
° Unused portions of the logbooks must be lined-out or "Z-ed" out.
° Logbook entries must be made in chronological order.
° For US EPA/CLP samples, entries are recorded for only one SDG on a page, except in the event where SDGs share quality control samples.
° Information inserted in the logbooks must be permanently affixed, signed, and dated across the insert.
° Copies of all pertinent logbook pages must be placed in the workorder file.
° Proper subsampling techniques can be located in the Subsampling SOP, 110.0039.

8.5.3 Extracts/Digestates:

The organic sample extracts, once completed by the preparation laboratory, are transferred to the appropriate instrumentation lab and signed into the extract transfer logbook. The analyst will sign for the extracts and enter storage location information. Inorganic metals and mercury digestates are transferred to the instrumentation lab. The analyst signs off on the preparation logbook at time of transfer.

When the time period for organic extract storage has expired, extracts may be disposed of in the same manner as would used/expired standards. CLP extracts must be stored for one year. The transfer and disposal for these extracts must be documented in the Extract Disposal Logbook.

When the time period for inorganic digestate storage has expired, digestates are disposed in the same manner as would used/expired inorganic standards.

See Mitkem SOP No. 30.0024 Sample and Waste Disposal for details on the actual disposal of above extracts/digestates.

8.5.4 Sample Disposal:

The unused portion of sample is returned to the Receiving area for long term storage. Such portions must be stored for a certain length of time as required by client contract and then properly disposed. Unused sample is disposed with the use of a licensed hazardous waste contractor. Routinely, one of the sample custodians runs the LIMS Omega Disposal program to produce a list of samples ready for disposal. See Mitkem SOP No. 30.0024 Sample and Waste Disposal for details on the different waste streams and disposal options.

All soil samples received from outside of the 48 contiguous United States must be disposed of following special procedures outlined by the USDA, as detailed in Mitkem SOP No. 30.0024 Sample and Waste Disposal section 8.1.4 Lab Solids, Broken Glass, Methylene Chloride, Acetone.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

10.1 The Sample Custodian will calibrate the temperature gun using an NIST calibrated thermometer on a quarterly basis. The correction factor will be noted on the temperature gun and recorded in the NIST Thermometer Calibration Logbook. The serial number of the NIST thermometer will also be recorded in the logbook.

10.2 On a daily basis, the Project Managers will review that the Sample Condition Forms are filled out correctly.

10.3 Multiple times daily, the temperatures of the refrigerators and freezers will be checked and the temperatures recorded electronically. Deviations will be noted in the electronic logbook and appropriate personnel notified for corrective action. If after 36 hours the temperature problem cannot be corrected with manual adjustments, the refrigerator or freezer will be replaced.

10.4 The Hazardous Waste Inspection Logbook will be completed weekly, after an inspection of the Hazardous Waste shed by a Sample Custodian. Major problems will be reported to the Hazardous Waste Coordinator.

11. Data Validation and Reporting

A copy of the chain of custody and Sample Condition Notification form accompanies the data reports to the client. Also any communication records are to be included in the data package (telephone conversations, e-mail communication, etc.).

12. Corrective Action Procedures

Corrective actions to be implemented in the event QC results are outside of the acceptance range. Notification reports (orange sheets) are generated in the event of an out-of-control situation occurs at sample log-in that cannot be corrected. Project Managers contact the client in order to ensure what type, if any, corrective action needs to be taken. Other corrective actions concerning the Receiving Area are recorded using the LIMS Corrective Action Report.

13. Health and Safety

The sample custodian must wear a lab coat, safety glasses, and gloves when handling samples and preservative chemicals. Samples coolers should be opened under a hood. All samples should be handled as though they are potentially hazardous. The outside of sample containers should also be handled as though they are contaminated. Any broken sample containers must be handled very carefully to prevent exposure to the sample.

14. Pollution Prevention, Waste Management, and Abbreviations

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

US EPA CLP SOW SOM01.2
US EPA CLP SOW ILM05.4
US EPA CLP SOW ISM01.2
US ACOE Shell for Chemical Analytical Requirements, Appendix H
Mitkem Quality Assurance Plan

Attachments:

1. **Attachment 1**: Mitkem Sample Condition Form.
2. **Attachment 2**: EPA Sample Condition Form (DC-1).
3. **Attachment 3**: Sample Condition Notification Form.
4. **Attachment 4**: Instructions for the use of pH paper.
5. **Attachment 5**: Example Authorized Personnel list.
6. **Attachment 6**: Sample Receipt and Tracking Logbook.
7. **Attachment 7**: VOA Receiving Logbook

Attachment 1
Mitkem Sample Condition Form

MITKEM LABORATORIES
Sample Condition Form

Received By:	Reviewed By:	Date:	Mitkem Work Order #:
Client Project:		Client:	

		Lab Sample ID		Preservation (pH) HNO_3	H_2SO_4	HCl	NaOH	H_3PO_4	VOA Matrix	Soil Headspace or Air Bubble ≥ 1/4"
1) Cooler Sealed	Yes / No									
2) Custody Seal(s)	Present / Absent									
	Coolers / Bottles									
	Intact / Broken									
3) Custody Seal Number(s)										
4) Chain-of-Custody	Present / Absent									
5) Cooler Temperature										
IR Temp Gun ID										
Coolant Condition										
6) Airbill(s)	Present / Absent									
Airbill Number(s)										
7) Samples Bottles	Intact / Broken / Leaking									
8) Date Received										
9) Time Received										
Preservative Name/Lot No.:										

VOA Matrix Key:

US = Unpreserved Soil	A = Air
UA = Unpreserved Aqueous	H = HCl
M = MeOH	E = Encore
N = NaHSO4	F = Freeze

See Sample Condition Notification/Corrective Action Form yes / no

Rad OK yes / no

Attachment 2

EPA Sample Condition Form (DC-1)

SAMPLE LOG-IN SHEET
FORM DC-1

Lab Name Mitkem Laboratories	Page 21 of 32
Received By (Print Name)	Log-in Date
Received By (Signature)	
Case Number	Sample Delivery Group No. / Mod. Ref. No.

Remarks: (1) Please see associated sample/extract transfer logbook pages submitted with this data package.

		EPA Sample #	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____ _____				
3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists	Present/Absent*				
4. Airbill	Airbill/Sticker Present/Absent*				
5. Airbill No.	_____ _____				
6. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain-of-Custody				
7. Sample Condition	Intact/Broken*/ Leaking				
8. Cooler Temperature Indicator Bottle	Present/Absent				
9. Cooler Temperature	_____				
10. Does information on TR/COCs and sample tags agree?	Yes/No*				
11. Date Received at Laboratory	_____				
12. Time Received	_____				
Sample Transfer					
Fraction (1) TVOA / VOA	Fraction (1) SVOA/Pest/Aroc				
Area #	Area #				
By	By				
On	On				

* Contact SMO and attach record of resolution.

Reviewed By	Logbook No.
Date	Logbook Page No.

Attachment 3
Sample Condition Notification Form

Sample Condition Notification

Mitkem Project#:_____ Date of Receipt:_____
Client:_____ Received By:_____
Client project #/name:_____

Unusual Occurance Description:

_____

_____

_____

_____

_____

_____

Client Contacted:
Contacted via: Phone/Fax/E-mail
Date:_____ Time:_____
Contacted By:_____
Name of person contacted:_____

Client Response:
Responded via: Phone/Fax/E-mail
Date:_____
Name of person responding:_____
Responding to:_____

_____

_____

_____

_____

_____

Mitkem Action Taken: _____

_____

_____

_____

_____

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Attachment 4

Instructions for use of pH paper

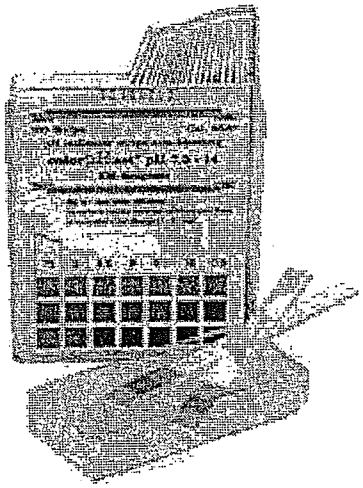
General Procedure for the Use of colorpHast® pH Test Strips

EMD

1. Remove a strip from the box.
2. Immerse the reaction zone of the strip into the solution for approximately 30 seconds to 1 minute, allowing enough time for the reaction to take place.
3. Remove the strip from the solution, wiping the strip along the edge of the vessel to remove excess liquid from the strip.
4. Compare the strip's reaction zones to the color chart on the box while holding the strip to the top of the box and the reaction zones to the bottom of the box.
5. Record the results of the closest matching color to the strip.

- *For additional application or product information, ordering information or other technical questions, please call our Technical Service Department at:*

  1-800-222-0342 *or* **1-856-423-6300**



EMD Chemicals Inc.
480 South Democrat Road
Gibbstown, NJ 08027

Phone: 856-423-6300
Fax: 856-423-4389
www.emdchemicals.com

Attachment 5

Example Authorized Personnel list

Sample log-in/out: **Authorized Personnel**		
Name	**Dept**	**Initials**
Anderson, Courtney	Inorganics	*CJA*
Appolonia, Gary	Inorganics	*GMD*
Badura, Karolina	PEST/PCB	*KB*
Cardoso, Antonio	Oprep	*AC*
Datta, Avijit	Receiving	*AED*
Ding, Yihai	Mitkem	*YD*
Huntley, Agnes	Project Management	*ARH*
Kaczorowski, Przemyslaw	Oprep	*PK*
Lawler, Edward	Project Management	*EL*
Lawler, Sharyn	QA/QC	*SBL*
Luo, Wei	VOA	*WL*
Lucas, Derek	PEST/PCB	*DL*
Maczewska, Beata	SVOA	*BM*
Marquis, Ashley	VOA	*ALM*
Thomson, Cassandra	Inorganics	*CT*
Mosher, Cassie	SVOA	*CLM*
Montmarquet, Timothy	Mitkem	*TM*
Ng, Shirley	Project Management	*SN*
Rosadzinski, Tomasz	Oprep	*TR*
Scarpaci, Matthew	SVOA	*MMS*
Sawyer, Tom	Inorganics	*TS*
Smart, Dawne	Inorganics	*DES*
Wetherbee, John	Inorganics	*JRW*
Veilleux, Brian	Oprep	*BV*
Wieczorek, Agnieszka	PEST/PCB	*AW*
Zharkova, Sofya	VOA	*SZ*
Zhao-Anderson, Huiyan	Inorganics	*HZA*

Most common personnel using log-in/out

Attachment 6

Sample Receiving and Tracking Logbook

MITKEM LABORATORIES, A Division of Spectrum Analytical, Inc.

Sample Receiving Logbook

Workorder No. _____

Client Name: _____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

OUT		IN	
Relinquished By	**Received By**	**Relinquished By**	**Received By**
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			

Comments: _____

_____

Please record analyst's initials, date, and sample #s removed. Add any comments if necessary (broken bottles, empty jars, etc.)
Include the abbreviated name of the test to be performed., ie: SVOA, PCB...near the "samp. #s".
Include bottle or jar number when more than one.

Reviewed:_____

Logbook ID: 30.0287-09/08

Attachment 7

Volatiles Receiving Logbook

MITKEM LABORATORIES: VOLATILE SAMPLES RECEIVING LOGBOOK

VOA Log-In Date	Workorder	Client ID	Sample Numbers	Relinquished by:	Received by:	Pres. Used	F/R	Returned to R1

Logbook ID 90.0191-10/09

Reviewed By:

"Preservative Used" Key

UA = Unpreserved Aqueous	H = HCL	A = Air	M = MeOH	E = *Encore
US = Unpreserved Soil	N = $NaHSO_4$		F = Freeze	T = Trace, HCL

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample and Waste Disposal

SOP No. 30.0024

Rev. 8

	Signature	Date
QA Director:		10/17/08
Lab Director:		10/21/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample and Waste Disposal

SOP 30.0024 Rev. 8

1. Scope and Application

This Standard Operating Procedure (SOP) outlines the procedures for the disposal of the unused portion of samples submitted for analysis, as well as solid and aqueous wastes and sample extracts to minimize the amount of waste in the lab and organize the manner in which it is handled.

2. Personnel Qualifications and Responsibilities

Personnel handling samples must be aware of the potential dangers posed by the samples, and handle them properly to minimize this danger. Personnel handling samples must also maintain the documented chain of custody of the samples from the time they are received at Mitkem until they become laboratory waste. Laboratory waste must be handled in a safe manner to minimize any exposure to this material.

3. Summary of Procedure

Waste stream manifest profiles have been identified by Mitkem and the waste-disposal company. These profiles dictate how to dispose of each individual waste, as described in this SOP. If any question arises regarding the disposal or classification of waste, the Waste Disposal Technician, or senior laboratory management should be consulted.

Once sample analyses are complete and a final report has been issued to the customer, samples are stored for a specified length of time before disposal. This timeframe is usually dictated by the client. Strict chain of custody documentation is maintained for these samples until samples are disposed into one of several waste streams. This waste is removed from Mitkem by a licensed waste-hauler for final disposal.

Safety equipment will be worn prior to and during any waste disposal. Proper gloves, safety glasses and lab coats or protective outerwear must be worn during all waste disposal activities.

4. Sample Preservation, Containers, Handling and Storage

Not applicable.

5. Interferences and Potential Problems

Not applicable.

6. Equipment and Apparatus

Waste drums. Polyethylene drums are used for liquids (acids or basic/spent CN solutions) and steel open top drums are used for waste solid materials.

Burp-free Ultra Funnel, various sizes, polyethylene funnel used to allow safe pouring of liquid wastes into drums. Product is vented and has locking lid.

Spill kits

Drum Pump

Drum Lift

Satellite Nalgene storage containers with lidded funnels for interim liquid waste storage in Wet Chem, Volatiles and Metals Labs.

Satellite storage containers (red metal Justrite Oily waste Can ,10G capacity) for interim solids storage in Organic Prep, Semivolatiles and Volatiles Labs.

Satellite storage container (Justrite Safety Disposal Can, 2G capacity) for solvent waste in Volatile Lab.

7. Reagents

Not applicable.

8. Procedure

8.1 Classification of Waste:

Laboratory waste products fall under one of the following categories:

8.1.1 Methylene Chloride, Methanol, Water:

Waste solvents are emptied into a solvent drum located in the organic sample preparation laboratory. This waste consists primarily of methylene

chloride with lesser portions of methanol and water. Trace amounts of other solvents may also be included from time to time. Due to the high proportion of methylene chloride in the waste, the material is flammable, and is classified as D001-type waste. Whenever a new drum is started, a Hazardous Waste label is attached to the outside.

When the drums are full the waste drum is taken to the Hazardous Waste storage shed. An accumulation date is added to the Hazardous Waste label on the drum.

Empty solvent bottles are evaporated to dryness and discarded into the general solid waste dumpster (garbage). Glass containers that present a risk of injury should be discarded into a broken glass container for disposal.

Any lab apparatus, gloves, paper towels, etc. exposed to methylene chloride must be discarded into the red solid waste drums in the Organic Prep Lab. The drum must be removed by the Waste Hauler within 90 days of the accumulation date.

8.1.2 Acids, Water:

Acid waste consists of approximately 98% water and 2% acid. This waste stream also includes the remainders of spent or expired metals standards, digested samples, TCLP and SPLP leachates and undigested aqueous samples preserved with acid. This waste is emptied into polyethylene acid waste drums in the hazardous waste area. Acidic waste from the Lachat analysis of nitrate/nitrite or chlorides are stored temporarily in Unit 3 in a small waste container equipped with a closing safety funnel. The container is routinely emptied into this same wastestream. A Hazardous Waste label is affixed to the side of the drum and an accumulation date is recorded on it the first time the drum is started. This waste is classified as D002 type waste. The drum must be removed by the Waste Hauler within 90 days of the accumulation date.

8.1.3 Scintillation Vials, Methylene Chloride, Hexane:

This waste stream consists of Organic analysis vials used for GC and GC/MS analyses. Spent or expired organic standards in solvent are also included in this wastestream. The vials are disposed of unopened. Vials are initially disposed in the GC Analysis Laboratory in a red metal drum. When this drum is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D001 waste.

8.1.4 Lab Solids, Broken Glass, Methylene Chloride, Acetone:

This waste stream contains the remainders of solid and soil samples. This waste stream also includes laboratory solid waste that has been stored in the red metal drums in the Organic Prep Lab. When full the laboratory red metal containers are taken to the Hazardous Waste shed and the contents dumped into the waste solids drum. A Hazardous Waste label with an accumulation date is affixed to the outside of the drum and is later removed by the Waste Hauler within 90 days of the accumulation date. This waste has no RCRA Characterization code.

Any solid matrix samples received from outside of the 48 contiguous United States, or from any other area identified by the client as a US Department of Agriculture Soil Quarantine Area must have special disposal in order to kill any quarantined organism. Samples identified as such, along with their entire container including jar cover, are placed in a disposable aluminum pan and placed in the kiln (in Unit 3) and heated to 400 degrees for 30 minutes. After the pan has cooled, the sample(s) and aluminum pan(s) are disposed in the waste soil/solid drum.See **Attachment 1** for incineration procedures.

8.1.5 Glass Vials, Methanol, Water, Soil:

This waste stream consists of primarily 40-ml organic analysis vials used for VOA analyses. The vials are disposed of unopened. Vials are initially disposed in the VOA Analysis Laboratory in a red metal drum. When this drum is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D001 waste.

8.1.6 Spent Carbon with Methylene Chloride:

This waste stream contains the remainders of Mitkem's discharge. Discharge is run through tanks of carbon material as a means of removing methylene chloride. When the tanks are fully deactivated, the carbon is removed and dumped into a steel drum. The tanks are then filled with activated carbon. A Hazardous Waste label with an accumulation date is affixed to the outside of the drum and the drum is later removed by the Waste Hauler within 90 days of the accumulation date. This waste stream is not associated with any RCRA Characterization code.

8.1.7 Electronic Device with Beryllium Oxide:

This waste stream contains the power tube of an ICP spectrophotometer. Because of beryllium in the tip of the tube, it cannot be disposed of with

ordinary trash. The tube is placed in a sealed cardboard box and a Hazardous Waste label with an accumulation date is affixed to the outside of the box. The box is later removed by the Waste Hauler. This waste stream is not associated with any RCRA Characterization code.

8.1.8 COD Vials:

This waste stream consists of glass tubes that contain a mixture of mercuric sulfate, silver sulfate, sulfuric acid, and chromic acid. The tubes are used for COD analyses and are disposed unopened in their entirety. Vials are initially disposed in the Wet Chemistry Laboratory in a covered cardboard box with a Hazardous Waste Label. When this box is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D002, D009, D011 waste.

8.1.9 Lab Pack Waste:

From time to time, waste or unused chemicals (non-solvents) will require disposal. These materials are segregated and placed in a lab pack container. An inventory of materials is maintained for the lab pack drum. The materials are stored in the laboratory in a closed cardboard box with a Hazardous Waste label attached. Consult the waste hauling company when sufficient volume of material is present in the laboratory to initiate a lab pack.

8.1.10 PCB Waste:

Any PCB-containing material (at a concentration of greater than 50 ppm) must be segregated and disposed according to PCB-specific procedures. This material would generally consist of any oil sample containing over 50 ug/g (ppm) of PCB. Samples containing this high concentration of total PCB will be identified by the GC Laboratory Manager or the Laboratory Director. The container for this sample will be prominently labeled "for return to client" when the holding period (as identified in **Section 8.2.1** below) is reached. The Mitkem project manager will be notified, as will the client, with a notation in the report Project Narrative or Cover Letter that the sample will be returned.

8.1.11 Water/Sodium Hydroxide/Pyridine:

This waste stream consists of sulfide and cyanide digestates and spent standards. Pyridine waste from the analysis of Cyanide (Lachat), and basic pH Ion Chromatography (IC1) wastes are stored temporarily in Unit 3 in several small waste containers equipped with closing safety funnels.

The containers are routinely emptied into a polyethylene Basic Waste drum in the hazardous waste area. This waste is classified as D002, D038 waste

8.2 Procedure for Disposal of Sample Remainders:

8.2.1 Determination of Disposal Date:

Samples are held for a period of time following receipt at the laboratory or release of the final report to the client. Mitkem's policy is to hold samples for a specified length of time. On a case-by-case basis, arrangements may be made between the client and Mitkem through discussion with the Mitkem Project Manager. This agreement is then saved in LIMS in the individual Client Project. Default values are used otherwise. To review the Sample Hold Times and Disposal Options on a particular workorder, go to the Report Option at the Workorder Main screen.

For CLP, samples are held for 60 days after delivery of the reconciled data package. Sample extracts must be stored at 4°C until 365 days after delivery of a complete, reconciled data package to EPA.

8.2.2. Documentation of Sample Disposal:

When the specified hold time has passed since the receipt date, the samples can be disposed. Sample receiving personnel select sample remainders from their storage locations and remove these to a designated location outside of the sample receiving area for disposal by Sample Receiving or laboratory personnel. Documentation that sample remainders have been disposed is maintained in the Mitkem Omega LIMS. When samples are removed from storage for disposal the person removing them enters the disposal date and their initials into LIMS.

8.2.3. Final Disposal and Record keeping:

All samples are to be disposed of according to **Section 8.1**.

Once waste drums are full, approximately bi-monthly, the waste hauling company is called to schedule a pick-up. Mitkem currently uses Univar to arrange for disposal of laboratory waste. Univar will remove the waste from the Mitkem storage area, and arrange with Pollution Control Industries (PCI) in Michigan for ultimate disposal of the waste material. Waste manifests are initiated by the hauler, signed by the QA Director or his designee when drums are loaded on the hauler's truck, signed and mailed back to Mitkem following final disposal. Manifests are filed

according to date in the QA office. Mitkem's Generator Number is **RI5000009621**.

9 Data Reduction and Calculations

Not applicable.

10 Quality Assurance/Quality Control

The Hazardous Waste Inspection Logbook will be completed weekly after an inspection of the Hazardous Waste shed by a Sample Custodian. Major problems will be reported to the Hazardous Waste Coordinator.

11 Data Validation and Reporting

After sample disposal, the disposal dates are entered into the LIMS for each workorder. In addition, the samples'storage location is changed to "disposed".

12 Corrective Action Procedures

Corrective actions concerning sample or waste disposal are recorded using the Omega LIMS Corrective Action Report under the Receiveing Department or another lab area involved in the issue. Corrective actions should implemented in the event of an out-of-control situation involving sample or waste disposal that cannot be easily corrected. The sample custodian should contact the Safety team, Hazardous Waste Coordinator or Lab Director for help determining what corrective action needs to be taken.

13 Health and Safety

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO THE USE of any chemical. In all cases both the applicable MSDS and supervisor or Safety Officer should be consulted. The employee should comply with all safety policies as presented in the Mitkem Safety Manual. Bottle or container labels also provide important information that must be noted. Proper gloves, safety glasses and lab coats or protective outerwear must be worn during all waste disposal activities.

All laboratory employees who either sign a Hazardous Waste manifest or handle hazardous waste directly must attend an annual RCRA training class. At this time Summit Technologies of New Hampshire provides the necessary on-site training for Mitkem's laboratory personnel.

14 Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15 References

None.

Attachments:

1. **Attachment 1**: Instructions for the Kiln.

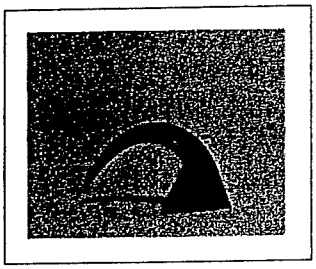
Attachment 1

Instructions for the Kiln

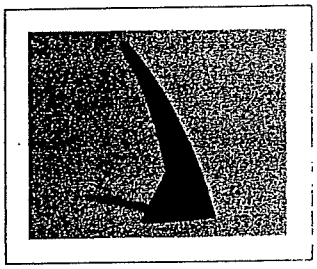
To use Cone-Fire Tuning, **IdLE** must display. Press **ENTER**, then **1. ConE** will appear. Press **999, ENTER. rSLt** (for result) will display alternating with the current Tuning number. Enter the new number, then press **ENTER. ConE** will appear. Continue entering the values for the Cone-Fire program in the usual way. Once you change the Tuning number, Cone-Fire will remain adjusted to that number until you change it again.

The large cone on the kiln shelf should be visible through a peephole. Avoid exposure to cool air by keeping the cone at least 3" away from the peephole. Program the DTC 800C for the cone on the shelf and fire. After cooling, check the cone:

The cone bent to 6 o'clock: In this case, the controller is matched to your kiln.

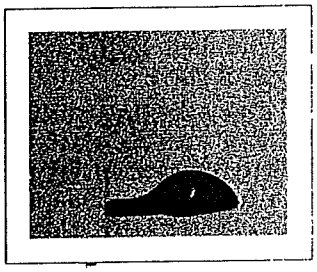


The cone did not bend far enough: Use a lower Tuning number for the next firing.



The cone bent too far: Use a higher Tuning number for the next firing.

Do not be overly concerned with achieving an exact 6 o'clock bend. The difference between a 3 o'clock and a 6 o'clock bend is only a few degrees. Cones, even from the same box, also vary slightly.



Ramp-Hold Mode

Ramp-Hold is available on both the DTC 800C and DTC 800.

In Ramp-Hold, the kiln fires in segments, or stages. Each segment has a firing rate, firing temperature, and hold (or soak) time. Ramp-Hold includes eight segments. Use only the number of segments you need per firing, from one to eight.

Rate is degrees of temperature change per hour, from 1° to 9999°F. (e.g., to increase temperature by 500° per hour, enter a rate of 500).

To control cooling, set the segment to a lower temperature than that of the preceding segment.

Storing User Programs in Ramp-Hold

A user program is a set of firing instructions to run the kiln in Ramp-Hold. A user program includes number of segments needed; firing rate, temperature, and hold (if any) for each segment; and an alarm temperature (if any).

Ramp-Hold Programming

As the program prompts you for segments, rate, temperature, etc., you will see values from the last firing. To use these again, just touch **ENTER**.

To fire without the Delay feature: Follow steps 1 through 9. Then press **START** twice.

1 Turn controller safety switch ON.

2 If controller displays **ErrP** or a flashing temperature, touch **ENTER. IdLE** will appear.

3 Touch **ENTER** then **4. USEr** will appear. Enter a number from 1 to 6 for the stored program desired. ENTER 1

4 Touch **ENTER. SEGS** will appear. Enter number of segments you will use. ENTER 1.

5 Touch **ENTER. rA 1** will appear. Enter firing rate for segment 1 (temperature change per hour: any temperature from 1° to 9999° F.). ENTER 400°C.

6 Touch **ENTER. °F 1** will appear. Enter the temperature you will be firing to in segment 1. ENTER 250°C

7 Touch **ENTER. HLd1** will appear. Enter segment 1 hold (soak) time in hours and minutes (e.g. 12 hours and 30 minutes = 12.30). ENTER 00.20

8 Touch **ENTER.** Continue entering values for all segments.

9 Touch **ENTER. AlAr** will appear. Enter alarm temperature. (Enter 9999 to turn alarm off.) Then touch **ENTER.**

IdLE will appear.

10 To set Delay Fire, touch **ENTER** then **3. dELA** will appear. Enter delay time in hours and minutes (e.g. 12 hours and 30 minutes = 12.30). Then touch **ENTER.** (Delay zeroes out after each completed firing.) NO DELAY

11 To start program, touch ENTER twice. **-On-** will appear, then kiln temperature. If a delay was programmed, **-On-** will appear, then time remaining until start.

To stop the program during the firing cycle, touch **STOP** or turn the safety switch OFF. When program fires to completion, **CPLt** will appear alternating with total firing time in hours and minutes. Turn safety switch OFF when **CPLt** appears. To shut off the alarm when it sounds during a firing, press **ENTER.** When Temp decreases after the 20 minutes @ 250°C, hit STOP. LET COOL DOWN ON ITS OWN.

Ramp-Hold can store up to six user programs even when the kiln is unplugged. Potters may want to store a program for crystalline glaze, another for a favorite red glaze, etc. Glass artists might want to store several programs for fusing their favorite types of glass and another for slumping.

When you enter Ramp-Hold mode, the first prompt to appear is **USEr.** The **USEr** prompt means, "Select a stored user program for this firing". If you are using Ramp-Hold for the first time, press 1. Then enter num-

ADDENDUM TO COMPLIANCE AGREEMENT WITH MITKEM CORPORATION
DECEMBER 18, 1996
CONTINUED

7. "USDA" soil samples may not be shipped to other facilities unless such facility has a valid permit and compliance agreement for imported soil or a valid compliance agreement for domestic soil. (See Item No. 3 for a list of such facilities)

8. "USDA" soil samples may not be used as a growing medium for plants and for the isolation and/or culture of organisms imported with the soil.

9. It is further agreed that the PPQ Officer may conduct an inspection of the laboratory facility and records concerning soil shipments at any time during normal working hours. All reasonable recommendations of the PPQ Officer to eliminate the spread of plant pests will be followed. Failure to comply with any of the stipulations of this Compliance Agreement may result in its revocation and the facility's authority to handle foreign and domestic regulated soil.

ALL SOIL RESIDUES, THEIR STORAGE CONTAINERS, FILTERS, ETC. SHALL BE TREATED WITH ONE OF THE FOLLOWING SCHEDULES:

Used soil storage containers may be treated by incineration.

DRY HEAT: Soil to be spread in layers 0.5 inches in depth to ensure uniform heat penetration.

TEMPERATURE		EXPOSURE PERIOD
110 - 120.5 C	(230 - 249 F)	16 HOURS *
121 - 154 C	(250 - 309 F)	2 HOURS *
154.5 - 192.5 C	(310 - 379 F)	30 MINUTES *
193 - 220 C	(380 - 429 F)	4 MINUTES *
221 - 232 C	(430 - 450 F)	2 MINUTES *

* Do not start counting time until the mass reaches the required temperature.

STEAM HEAT: 250 F at 15 lbs. pressure for 30 minutes.

Preheat laboratory autoclaves. Do not start counting time until pressure reaches 15 lbs.. Restrict soil depth to 2 inches when treating quantities of soil in trays. Restrict each package weight to 5 pounds or less when treating individual packages. Load with adequate spacing. Large commercial steam facilities which operate at pressures up to 60 lbs. psi will permit treatment of greater soil depth.

For large filter & soil — use 250° for 2 hours

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Aqueous Samples by Separatory Funnel (Method 3510C)

SOP No. 50.0051

Rev. 2

	Signature	Date
QA Director:	Sharon B Lewis	2/2/10
Lab Director:	[illegible]	2/4/10

Effective Date: 02/11/10

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Aqueous Samples by Separatory Funnel (Method 3510C)

Rev. 2

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the operations for the preparation of aqueous samples using separatory funnel extraction. Discussions include sample extraction, sample cleanup references, and sample concentration technique of aqueous samples for analysis by SW-846 Methods 8081, 8082, 8270, 8015(DRO/TPH), MA EPH and EPA Method 625.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 1-liter sample is solvent extracted using a separatory funnel. The extract is dried through granular anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using a Caliper TurboVap apparatus or Kuderna-Danish (KD) apparatus, and nitrogen blow down.

4. Sample Preservation, Containers, Handling, and Storage

4.1. The sample holding times are as follows:

- Sample must be extracted within 7 days from the time of sample collection
- Sample extracts must be analyzed within 40 days of sample extraction.

4.1 Samples should be in 1-L amber glass jars.

4.2 Samples are stored at 4°C until time of extraction.

4.3 DRO samples are preserved in the field with H_2SO_4 to a pH < 2.

4.4 Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results for the analytes of interest. See Mitkem SOP Nos. 50.0030 through 50.0034 for cleanup methods.

5.3 Phthalate esters can contaminate sample extracts, and many products found in the laboratory contain these esters. Plastics in particular must be avoided during the preparation steps to minimize interferences and contamination from these compounds. Several phthalate esters are target compounds in the semivolatile analyses. Contamination of method blanks and samples with these phthalates poses additional quality issues with the resulting data.

6. Equipment and Apparatus

Instrumentation used in this preparation method include:

6.1 Separatory funnel – 2L, glass or Teflon.

6.2 Erlenmeyer flask – 500mL.

6.3 1L graduated cylinder.

6.4 Wide range pH strips, Whatman or equivalent.

6.5 Glass syringes for delivering lab control spike, matrix and surrogate spike solutions.

6.6 Volumetric flasks for making up surrogate and matrix spike solutions – 50mL, 250mL.

6.7 Kuderna-Danish apparatus with a 10mL or 15mL receiver tube.

6.8 Glass funnel.

6.9 Three ball Synder column.

6.10 Water bath, capable of maintaining 70 °C or 95°C.

6.11 Boiling chips, carbon.

6.12 Nitrogen blowdown apparatus, N-EVAP Model No. 111.

6.13 Receiving vial with Teflon septa – 2mL.

6.14 Glass Pipettes.

6.15 20X150mm Disposable Culture Tubes.

6.16 Turbo Vap Collection Tubes.

6.17 Caliper TurboVap

6.18 Aluminum Foil (Industrial Grade).

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. The following chemicals including solvent, standards and gasses are extensively used in the lab:

7.1 Methylene chloride: pesticide quality or equivalent, to be used for glassware rinsing and sample extraction.

7.2 Methanol: pesticide quality or equivalent, to be used for preparing standards.

7.3 Acetone: pesticide quality or equivalent, to be used for rinsing glassware.

7.4 1:1 v/v H_2SO_4 to be used for pH adjustment.

7.5 10N NaOH to be used for pH adjustment.

7.6 Anhydrous sodium sulfate, granular for drying the sample extract.

7.7 Surrogate standards: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatiles Full Scan: Restek BN Surrogate mix (Cat. No. 31086)at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Semivolatiles SIM: Cambridge Benzo(e) pyrene d-12 surrogate (Cat. No. DLM-257-S) at 200µg/mL.

- Deisel Range Organics (DRO)/TPH and MA EPH: 5-α Androstane at 10,000μg/mL (Made from neat source(Sigma)), and o-Terphenyl at 10,000μg/mL(Made from neat source(Aldrich)).

- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 μg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000μg/mL in Acetone.

7.8 Lab Control Sample and Matrix Spike: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000μg/mL in Methylene Chloride; Restek 3,3' Dichlorobenzidine (Cat. No. 31026) at 2000μg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000μg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
- DRO/TPH: Diesel/gasoline.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80μg/mL and Mirex (UltraScientific Cat. No.PST-720M100A01) at 100ug/mL. For the LCS/MS mix a 10.0ug/mL Intermediate standard is prepared for Mirex..
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000μg/mL in Hexane.
- MA EPH: An independent vendor than that used for the MA EPH calibration standards is used. The LCS consists of the 17 Aromatic Hydrocarbon compounds plus the 14 normal Aliphatic Hydrocarbons at 1000μg/mL in Methylene Chloride.

8. Procedure

8.1 Standards Preparation:

All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1 All primary standards received from vendors are logged into the Semivolatile or Pesticide/PCB Primary Standard Logbook. These include standards for surrogates, LCS and matrix spike. The standards are labeled ZPyymmddX, where:

Z= S for Semivolatile and DRO, P for Pest/PCB
P = Primary standard
yymmdd = date standard is received, and

X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2 Preparation of Surrogate standard:

Semivolatile Full Scan: The working surrogate standard is prepared by combining 2.5mL of the Base/Neutral primary standard and 1.075mL of the Acid primary standard in **Section 7.7** and diluting to 250mL using methanol. The working standard contains the following compounds:

Compound	Concentration(µg/mL)
1,2-Dichlorobenzene-d_4	50
2-Fluorobiphenyl	50
Nitrobenzene-d_5	50
p-terphenyl-d_{14}	50
2-Chlorophenol-d_4	75
2-Fluorophenol	75
Phenol-d_6	75
2,4,6-Tribromophenol	75

Semivolatile SIM: The working surrogate standard is prepared by diluting 1.25mL of the 200ug/mL primary standard in Section **7.7** to 50mL in methanol. The resulting concentration is 5ug/mL of Benzo(e) pyrene d-12.

DRO/TPH: The working surrogate standard is prepared by taking 1mL of the 5-α Androstane and 1.0 mL of the o-Terphenyl primary standards in **Section 7.7** and diluting to 200mL using methanol. The working standard contains the following compounds:

Compound	Concentration(µg/mL)
o-Terphenyl	50
5-α Androstane	50

Pesticide/PCB: The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.7** , and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

Compound	Concentration(µg/mL)
Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

The working surrogate spike standard is labeled ZWyymmddX where:

Z= S for Semivolatile and DRO/TPH, P for Pest/PCB
W = working standard
yymmdd = date working standard is prepared,
X = the order that the working standard is prepared on that date, in increasing alphabetical order.

A smaller or larger volume of the stock solution may be used to prepare the surrogate spike. The final volume of the surrogate solution will be adjusted accordingly to achieve the same spike working concentration.

The standard is placed in amber screw top bottles and stored in the freezer at –10 to –20°C. They are stored in a separate location from sample extracts to make sure that there is no sample and/or sample extract cross contamination.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3 Preparation of Lab Control Spike (LCS) and Matrix Spike (MS) Standard:

Semivolatiles Full Scan: The working Semivolatile LCS/MS Standard is prepared by combining 5.0 mL of 8270 MegaMix, 2.5mL of 3,3' DCB, and 5.0 mL of 8270 add-on intermediate standard in **Section 7.8** and diluting to 50mL using methanol. The working LCS/MS Standard contains the full list of SW8270 compounds at 50 µg/mL.

Semivolatile SIM: The working LCS/MS Standard is prepared by a further 10 times dilution of the above standard.

DRO/TPH: The working LCS/MS Standard is prepared by first preparing an intermediate standard and making a dilution of it.

On an analytical balance, weigh out 5.00g of diesel gasoline into a 10mL volumetric flask and bring to volume with Methanol. This is the intermediate standard. The working LCS/MS Standard is prepared by taking 1mL of the intermediate standard and diluting to 100mL using Methanol. The working LCS/MS Standard contains the following compounds:

Compounds	Concentration(µg/mL)
Diesel Gasoline	5000

Pesticides: The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2 and 1.0mL of Intermediate Mirex standard in **Section 7.8**, and diluting it to 50 mL in methanol. The working LCS/MS Standard

contains the full list of SW8081 individual response compounds. See Attachment 1 for a list of the individual compounds and their concentrations.

PCB: The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.8** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

Compounds	Concentration(µg/mL)
Aroclor 1016	4.0
Aroclor 1260	4.0

MA EPH :The working MA EPH LCS/MS Standard is prepared by taking 2.5mL of the Aliphatic Hydrocarbons standard in Section **7.8** and 2.5mL of the Aromatic Hydrocarbon standard in Section **7.8** and diluting to 250mL in 1:1 methylene chloride: acetone mixture . The final concentration will be 50 ug/ml for each of the individual components.

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2**.

A smaller or larger volume of the stock solution may be used to prepare the LCS/MS Standard. The final volume will be adjusted accordingly to achieve the same working concentration.

The standard is placed in amber screw top bottles and stored in the freezer at –10 to –20°C. They are stored in a separate location from sample extracts to make sure that there is no sample and/or sample extract cross contamination.

The expiration date for the ampulated solutions is discussed in **Section 8.1.1.**

All of the appropriate standard preparation information is to be recorded in the working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2 Sample Extraction Procedure:

Aqueous samples are solvent extracted using a 2-L separatory funnel. The glass separatory funnels must be inspected periodically to ensure that there are no cracks. The Teflon stoppers must also be inspected to make sure there are no scratches. The Teflon separatory funnels must be inspected periodically to ensure there are no scratches.

8.2.1 The aqueous sample to be extracted is allowed to warm to ambient temperature prior to extraction. A representative 1L portion of the sample is measured by first marking the sample level on the bottle (which is not a Mitkem bottle). The contents of the container are poured into the separatory funnel. The marked bottle

is then used to measure the sample. If the sample bottle is a Mitkem bottle, a bottle for each lot number will be calibrated and marked. This bottle will be used as a reference when determining initial sample volume. An alternate option is to use a clean 1 L Class A graduated cylinder to measure the sample.

8.2.2 The sample pH is taken and pH adjusted to between 5 – 9 if necessary. Add 1:1 H_2SO_4 or 10N NaOH as needed to the sample such that pH is between 5 – 9. The 1L aliquot is then transferred to the separatory funnel. The pH may be taken using a clean glass stir rod or a glass pipette. Another option is to open the stopcock on the sample for a moment and allow a drop or two to drop onto the pH paper directly from the separatory funnel. This must be done BEFORE the addition of solvent. For semivolatile extractions, the sample is acidified to a ph<2. DRO samples should have been acidified in the field. Acidify any DRO/TPH samples which were not field preserved. Document the pH adjustment of any Pesticide/PCB and DRO/TPH samples in the associated extraction logbook.

8.2.3 The graduated cylinder or sample bottle is rinsed with 60mL of methylene chloride, which is also added to the separatory funnel. Use 1L of organic free water is necessary for the Blank and LCS.

8.2.4 Sonicate the surrogate standard and the lab control sample standard for about 10 minutes.

- Use a syringe to add 1.0 mL of the surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in a separatory funnel. The standards should mix with the aqueous sample immediately since the solvent is methanol.
- Use another syringe to add 1.0 mL of the lab control sample standard into the LCS and any MS/MSD samples.

8.2.5 Transfer the rinsate from the graduated cylinder or the sample bottle, in **Section 8.2.3** to the separatory funnel. The bottle is saved for volume determination. For the Blank and LCS, add 60mL of methylene chloride to the separatory funnel.

8.2.6 Stopper the separatory funnel and invert. Vent the separatory funnel immediately to release the built up vapor. Shake the separatory funnel for 3 minutes, being sure to vent periodically to release the built up vapor.

8.2.7. At the end of the extraction, the separatory funnel is allowed to settle with the stopper removed. The separatory funnel should be allowed to sit undisturbed for at least 10 minutes to allow for phase separation. If an emulsion develops, use any mechanical means, i.e. glass rod, available to break up the emulsion. If emulsions persist and do not break up with mechanical means, ask the supervisor for further instructions(may include preparation using continuous Liquid/ Liquid extraction).

8.2.8. Decant the bottom methylene chloride layer into a 500mL flask.

8.2.9. Repeat the extraction two more times with 60mL of methylene chloride each time.

8.2.10. Decant the bottom methylene chloride layer to the 500mL flask with care to limit the amount of water in the extract.

8.2.11. For Semivolatile sample extraction, the sample is then made basic with 10N NaOH to a pH > 10. Add 60mL of methylene chloride and shake for 3 minutes venting periodically.

8.2.12. Decant the bottom layer of methylene chloride into the 500mL flask. Repeat the extraction 2 more times.

8.2.13. Cover the extracts with aluminum foil until sample extract concentration.

8.2.14. Record all extraction information in the Prep Lab bench sheet (**Figures 1 and 2**).

8.3. Sample Filtration and Concentration:

The sample extract is now ready for filtration and concentration. These procedures are found in SOP No. 50.0054, Extract Filtration and Concentration. Cleanup procedures can be found in SOP No. 50.0030 through 50.0034.

8.4. The extracts are transferred to the Semivolatile lab and documented in the appropriate Extract Transfer Log. The extracts are stored in the refrigerator at 4°C until analysis.

8.5. Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

8.5.1. Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of.

8.5.2. Sample Extracts – All sample extracts are kept for 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of.

9. Data Reduction and Calculations

9.1 Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{\text{(Concentration of ampule)(amount used)}}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab

control sample, matrix spikes and balance checks. In order to trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and contain the manufacturer's reference number given in the standard preparation log book.

10.1 Method Blank – A method blank is a volume of organic free reagent water that is carried through the entire analytical procedure. It is used to determine the level of contamination associated with the analytical processing and analysis of samples.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank:

- The Method Blank is prepared in identical fashion as the associated samples.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled MB and is assigned a batch number in increasing order for each new batch of twenty samples or less.

10.1.3 Acceptance criteria for Method Blank:

All samples associated with a non-compliant Method Blank are re-extracted and reanalyzed. See the associated analytical SOP.

10.2 Surrogate – Surrogate standards are added to all samples including the Method Blank, Lab Control Sample and matrix spikes to assess the efficiency of the sample preparation and analysis procedures.

10.3. Lab Control Sample (LCS) – A Lab Control Sample is a volume of organic free reagent water that is spiked with all the target analytes and surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1. Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.3.2. Procedure for LCS:

- The LCS is prepared in identical fashion as the associated samples; in addition the appropriate amount of surrogate solution prepared in **8.1.2** and appropriate amount of the lab control spike prepared in **8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled LCS and is given the corresponding batch number that is associated with the method blank.

Any sample(s) that is/are associated with a non-compliant LCS will require re-extraction and re-analysis. See the associated analytical SOP.

10.4. Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.4.1. Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Once every 20 samples, or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.4.2. Procedures for Duplicate Matrix Spikes:

- The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition
- 1.0mL of the surrogate solution prepared in **8.1.2** and 1.0mL of lab control spike prepared in **8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.

11. Data Validation and Reporting

11.1 The supervisor will review data generated in the organic preparation laboratory. The Quality Control Officer will perform periodic and unscheduled reviews. These data consist of the standards preparation, volume of samples, and the volume of solvent used.

11.2 Reporting of the data will a include review by the Organic Preparation Laboratory Supervisor of the data listed in **11.1**, as well as time of extraction, sampling handling procedures, and extract handling procedures.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1. Method Blank Analysis:

All samples associated with a non-compliant Method Blank are re-extracted and reanalyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when method blanks have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out (**Figure 3**). The re-extracted samples will be labeled with the suffix RE.

12.2. Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out (**Figure 3**). The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects.

12.3. LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. Any sample(s) that is/are associated with a non-compliant LCS will require re-extraction and re-analysis. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out (**Figure 3**).The re-extracted samples will be labeled with the suffix RE.

12.4. Matrix Spike Recovery and RPD

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan: Mitkem Laboratories, A Division of Spectrum Analytical, Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

Attachments:

Attachment 1: Pesticide MS/LCS list
Figure 1: Semivolatile Extraction Logbook
Figure 2: Pesticides/PCB Extraction Logbook
Figure 3: Re-extraction Logbook

Attachment 1: Pesticide MS/LCS list

Analyte	Spike conc in ug/mL
4,4´-DDD	0.4
4,4´-DDE	0.4
4,4´-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2
Mirex	0.2

Figure 1: Semivolatile Extraction Logbook

MITKEM LABORATORIES **ORGANIC PREP - SAMPLE PREPARATION: SEMIVOLATILES**

Date:	Analysis:	Method #	Aq: 3510C (SepF) 3520C (Liq/Liq) Other: Soil: 3550B (Sonic) 3540C (Soxhlet) _____	Matrix Aqueous Soil Wipe Oil Other:_____	Sodium Sulfate Lot #:

Batch ID	LCS ID	Analyst	Spiked By	Witness	MeCl Lot#	Acetone Lot#	H_2SO_4 Lot#	NaOH Lot#	Time/Date Started:
									Time/Date Ended:

Lab ID	Bottle #	Sample Wt (g) / Vol (mL)	Initial pH	Surrogate Spike Added(uL)	Matrix Spike Added(uL)	H2SO4 pH<2	NaOH pH>11	Emulsion	KD/RV Prior to GPC or Fractionation Date / Analyst		GPC or Fractionation Date / Analyst		Final Concentration Date / Analyst		Final Conc. Volume (mL)	Date Extract Trans.

Comments:

Logbook ID: 50.0147-01/10

Surrogate Std. ID:

Matrix Spike/LCS Std. ID:

Sonicator Tuned: Yes / No

Soxhet Cycle/Hour:_____

Drip Rate_____

Water Bath Temp:_____

Reviewed By:_____

Figure 2: Pesticides/PCB Extraction Logbook

MITKEM LABORATORIES ORGANIC PREP-SAMPLE PREPARATION:PEST/PCB

Date:	Prep Method	Aq: 3510C (SepF) 3520C (Liq/Liq) Soil: 3550B (Sonc) 3540C (Soxhlet) 3570(MSE) Other:_____	Matrix: Aqueous Soil Wipe Oil Other:_____	BATCH ID:	Date/Time Started:

Analyst	Spiked By	Witness	Solvent: Lot #	GPC Batch Number	Florisil Lot #	Analysis:	Date/Time Ended:

	Lab ID	Bottle #	Sample Wt (g) / Vol (ml)	Initial pH	Emulsion?	Initial CONC Date/ Analyst TV/ KD	Extract volume (ml)	GPC Cleanup Date / Analyst	FLORISIL Cleanup Date / Analyst	ACID (PCB only) Cleanup Date / Analyst	SULFUR (Copper) Cleanup Date / Analyst	Final CONC Date/ Analyst TV/ KD	Final extract volume (ml)	Extract Transfer Date /By

Comments:

PEST Matrix Spike Std ID:	Vol. Spiked:		
PCB Matrix Spike Std ID:	Vol. Spiked:		
PEST Surrogate Spike Std ID:	Vol. Spiked:	Sodium Sulfate lot#	
PCB Surrogate Spike Std ID:	Vol. Spiked:	Hexane lot #	L/L Drip Rate:
PEST LCS Spike Std ID:	Vol. Spiked:	Sulfuric Acid lot#	Soxhlet Cycle/Hour:
PCB LCS Spike Std ID:	Vol. Spiked:	Sulfur cleanup Copper lot #	Sonicator Tuned? Yes/No

Figure 3: Re-extraction Logbook

Mitkem Laboratories, A Division of Spectrum Analytical, Inc.

RE-EXTRACTION LOGBOOK
ORGANICS

REQUESTED BY:
DATE:

Mitkem Sample ID	Sample Matrix	Analysis	MB originally associated with sample	Reasons for Re-extraction

PREP LAB APPROVAL:

DATE:

Logbook ID 50.0190-06/08

Reviewed By:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Sonication (Method 3550B)

SOP No. 50.0052
Rev 3

	Signature	Date
QA Director:	Sharon B Lewis	2/2/10
Lab Director:	[illegible]	2/4/10
Effective Date:	02/11/10	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Sonication
(Method 3550B)
Rev 3

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil and sediment samples using ultrasonic extraction for analysis by EPA SW846 methods SW8081, SW8082, SW8015 and SW8270. Discussions include sample extraction, sample cleanup references, and the preparation of standard spiking solutions for the analysis of Total Petroleum Hydrocarbons (TPH), Diesel Range Organics (DRO), pesticide/PCB or semivolatile organic compounds in soil samples. Highly contaminated soils for MA DEP EPH analyses may also use this method.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts in the Organic Preparation Laboratory (OPREP). Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 30-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. The sample is solvent extracted three times using ultrasonic extraction. The extract is separated from the sample by gravity filtration. The extract is dried through powdered anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using TurboVap or Kuderna-Danish (KD) apparatus followed by Nitrogen blowdown. In addition, this method can be used for Medium/High Concentration Semivolatile samples. A 1-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. This is solvent extracted once using ultrasonic extraction. The extract is separated from the sample by gravity filtration. The extract is dried through anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration.

4. Sample Preservation, Containers, Handling, and Storage

4.1. The sample holding times are as follows:

- Sample must be extracted within 14 days from the time of sample collection.
- Sample extracts must be analyzed within 40 days of sample extraction.

4.2. Sediment/Soil samples – Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Refer to **SOP No. 110.0039** for more detail on sub sampling techniques.

4.3. Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

5.1. Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2. Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results of analysis of the analytes of interest.

5.3. Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

6. Equipment and Apparatus

Instrumentation used in this preparation method include:

6.1. Ultrasonic Disrupter – pulsing horn type with 375 watts maximum and ¾" standard solid disrupter horn and ⅛" standard tapered microtip probe(medium soils only). Fisher Scientific Sonic Dismembrator Models 500 and 550.

6.2. Sonabox – ultrasonic disrupter box designed to reduce exposure to ultrasonic sound.

6.3. Beakers – 400mL

6.4. 500mL Erlenmeyer flask

6.5. Dessicator

6.6. Balance capable of weighing +/- 0.1 gram.

6.7. Glass syringes for delivering spike and surrogate solutions – 0.25mL, 0.5mL, 1.0mL.

6.8. Volumetric flasks for making up surrogate and matrix spike solutions – 50mL, 250mL.

6.9. Aluminum Foil (Industrial Grade)

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. The following chemicals including solvents and standards are extensively used in the lab:

7.1. Methylene chloride: pesticide quality or equivalent to be used for glassware rinsing and sample extraction.

7.2. Methanol: pesticide quality or equivalent, to be used for rinsing glassware and preparing standards.

7.3. Acetone: pesticide quality or equivalent, to be used for rinsing glassware.

7.4. 1:1 v/v methylene chloride/acetone mixture, to be use for sample extraction.

7.5. Hexane: pesticide quality or equivalent , to be used for solvent exchange of samples.

7.6. H_2O, deionized.

7.7. H_2SO_4, concentrated, for sample pH adjustment.

7.8. Surrogate Standards *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatiles Full Scan: Restek BN Surrogate mix (Cat. No. 31086)at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Semivolatiles SIM: Cambridge Benzo(e)pyrene d-12 surrogate (Cat. No. DLM-257-S) at 200µg/mL.
- Deisel Range Organics (DRO)/TPH and MA EPH: 5-α Androstane at 10,000µg/mL (Made from neat source(Sigma)), and o-Terphenyl at 10,000µg/mL (Made from neat source(Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.

7.9. Lab Control Sample and Matrix Spike: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3,3' Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- TPH/DRO: LCS uses Diesel/gasoline.
- MA EPH: An independent vendor than that used for the MA EPH calibration standards is used. The LCS consists of the 17 Aromatic Hydrocarbon compounds plus the 14 normal Aliphatic Hydrocarbons at 1000µg/mL in Methylene Chloride.

7.10. Anhydrous sodium sulfate: granular for drying the samples and sample extracts. Baked at 400°C for four hours .

8. Procedure

8.1. Standards Preparation: All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1. All **primary standards** received from vendors are logged into the Semivolatile or Pesticides/PCB Primary Standard Logbooks. These include standards for surrogates, LCS and matrix spikes. The standards are labeled ZPyymmddX, where:

Z = S for Semivolatile or TPH/DRO, P for Pesticides or PCB
P= Primary standard
yymmdd = date standard is received, and
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2. Surrogate standard:

8.1.2.1. Semivolatile Full Scan: The Semivolatile working surrogate standard is prepared by combining 2.5mL of the Base/Neutral primary standard in **Section 7.8** and 2.5mL of the Acid primary standard in **Section 7.8** and

diluting to 250mL using methanol.

The working standard contains the following compounds:

Compound	Concentration(µg/mL)
1,2-Dichlorobenzene-d_4	50
2-Fluorobiphenyl	50
Nitrobenzene-d_5	50
p-terphenyl-d_{14}	50
2-Chlorophenol-d_4	75
2-Fluorophenol	75
Phenol-d_6	75
2,4,6-Tribromophenol	75

Semivolatile SIM surrogate standard is prepared by diluting 1.25mL of the 200ug/mL primary standard in **Section 7.8** to 50mL in methanol. The resulting concentration is 5ug/mL of Benzo(e) pyrene d-12.

The working surrogate spike standard is labeled SWyymmddX where:
SW = Semivolatile working standard
yymmdd = date working standard is prepared,
X = the order that the working standard is prepared on that date, in increasing alphabetical order.

8.1.2.2. The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.8** , and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

Compound	Concentration(µg/mL)
Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

8.1.2.3. The TPH/DRO and EPH working surrogate standard is prepared by taking 1mL of the 5-α Androstane and 1mL of the o-Terphenyl primary standards in **Section 7.8** and diluting to 200 mL using methanol. The working standard contains the following compound:

Compound	Concentration(µg/mL)
5-α Androstane	50
o-Terphenyl	50

A smaller or larger amount may be used to prepare the surrogate. The final volume of the surrogate solution will be adjusted accordingly.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3. Lab Control Sample (LCS) and Matrix Spike (MS) Standard:

8.1.3.1. The Semivolatile Full Scan working LCS/MS Standard is prepared by combining 5.0 mL of 8270 MegaMix, 2.5mL of 3,3' DCB, and 5.0 mL of 8270 add-on intermediate standard from **section 7.9**, and diluting to 50mL using methanol. The resulting concentration will be 50ug/mL for all SW8270 compounds. The working Semivolatile SIM LCS/MS Standard is prepared by a further 10 times dilution of the above standard.

8.1.3.2. The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2, and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds at 2-20 µg/mL. See **Attachment 1** for a list of the individual compounds and their concentrations.

8.1.3.3. The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.9** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

Compounds	Concentration(µg/mL)
Aroclor 1016	4.0
Aroclor 1260	4.0

8.1.3.5. The TPH/DRO working LCS/MS Standard is prepared by first preparing an intermediate standard and making a dilution of it.

On an analytical balance, weigh out 5.00g of diesel fuel into a 10mL Class A volumetric flask and bring to volume with Methanol.

The intermediate spike standard is labeled SIyymmddX
where:
SI = Semivolatile intermediate standard
yymmdd = date intermediate standard is prepared,
X = the order that the intermediate standard is prepared on that date, in increasing alphabetical order.

The working LCS/MS Standard is prepared by taking 1mL of the

intermediate standard above, and diluting to 100mL using Methanol. The working LCS/MS Standard contains the following:

Compounds	Concentration(µg/mL)
Diesel fuel	5000

The working MA EPH LCS/MS Standard is prepared by taking 2.5mL of the Aliphatic Hydrocarbons standard in Section **7.9** and 2.5mL of the Aromatic Hydrocarbon standard in Section **7.9** and diluting to 250mL in 1:1 methylene chloride: acetone mixture . The final concentration will be 50 ug/ml for each of the individual components.

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2.1.**

A smaller or larger amount may be used to prepare the Lab Control Sample and Matrix Spike Standard. The final volume will be adjusted accordingly.

The standard solution is placed in amber bottles and stored in the freezer in the GC or GC/MS lab at –10 to –20°C. One bottle is transferred to the OPREP Lab and stored in the freezer at –10 to –20°C. The bottles are stored in a separate location from samples or sample extracts to make sure that there is no cross contamination.

The expiration date for the ampulated solutions is discussed in **Section 8.1.1.**

All of the appropriate standard preparation information is to be recorded in the appropriate Lab's working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2. Sample Extraction Procedure:

8.2.1. Low Level Method: Unless Mitkem has documented history that the samples contain high concentration of target analytes (>10,000µg/Kg for the individual analyte) all of the semivolatile samples are prepared using the low-level approach.

Before starting:

- The disrupter has a minimum of 375 watts power and must be inspected periodically to ensure that the ¾ inch tip has not experienced excessive wear.
- The disrupter is tuned each day of use. Follow manufacturer's guidance for tuning procedures (see instructions posted by Sonicators). The tuning is

documented in the Sonicator Horn Tuning Logbook, **Figure 4**. After tuning, note in extraction logbook as well.

- Remove the surrogate and LCS/MS spiking standards from the freezer and allow them to reach room temperature.

8.2.1.1. The sample is mixed to ensure sample homogeneity. A representative portion of the sample is measured into a pre-weighed 400mL glass beaker. A 30 gram ± 0.5 gram sample is transferred into the beaker using a clean stainless steel scoopla. The weight measurement is recorded to the nearest 0.1 gram. Calibrate the balance prior to use. Refer to **SOP No. 110.0007** for direction.

8.2.1.2. Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. When mixed with the anhydrous sodium sulfate, the solid material must be a free flowing, however, it should not contain an excessive amount of anhydrous sodium sulfate. If a sample matrix presents difficulty, discuss the issue with the OPREP Supervisor or Mitkem's Technical Director before proceeding.

8.2.1.3. Sonicate the surrogate standard and the LCS/MS standard prepared in **Sections 8.1.2** and **8.1.3** for about 10 minutes. According to the test method being used, add the following spikes to the samples in beakers:

- Use a syringe to add 1.0mL of the surrogate standard to the Blank, LCS, samples and any MS/MSD sample.

- Use a syringe to add 1.0mL of the LCS/MS standard to the LCS, and any MS/MSD sample.

8.2.1.4 Add 100mL of the 1:1 v/v methylene chloride/acetone mixture to the Blank, LCS, samples, and any MS/MSD for Semivolatile or Pesticide/PCB extraction, OR add 100mL of methylene chloride to the Blank, LCS, samples, and any MS/MSD for TPH/DRO extraction.

8.2.1.5 Place the bottom surface of the ¾ inch tip of the disrupter about ½ inch below the surface of the solvent, but above the solid layer.

8.2.1.6 Extract ultrasonically for 3 minutes, with the output control set at full power and the mode switch on Pulse, using a 50% duty cycle.

8.2.1.7 Remove the beaker from the disrupter. Decant the solvent layer into a 500mL Erlenmeyer flask, leaving the solid in the beaker.

8.2.1.8 Extract the sample two more times with 100mL of solvent and a 3 minute sonication time. Decant the solvent aliquots into the 500mL Erlenmeyer flask. Cover the flask with aluminum foil.

8.3. Semivolatile Medium Level Method: (Also refer to **SOP No. 50.0100** , Method SW846 3570 Microscale Solvent Extraction for another soil extraction option using small sample volumes.)

8.3.1. The sample is mixed to ensure sample homogeneity. As representative as possible, weigh about a 1 gram portion of the sample into a pre-weighed 15mL vial. The weight measurement is recorded to the nearest 0.1 gram.

8.3.2. Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. Mix the sample and anhydrous sodium sulfate to achieve a free flowing mixture.

8.3.3. Use a syringe to add 1.0mL of the surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in the 40mL vial.

8.3.4. Use a syringe to add 1.0mL of the lab control sample standard into the LCS and any MS/MSD samples.

8.3.5. Add 9mL of methylene chloride to the Blank and samples. Add 8mL of methylene chloride to the LCS and any MS/MSD.

8.3.6. Sonicate the sample with the ⅛ inch tapered Microtip for 2 minutes at output control setting at 5 in the continuous mode.

8.3.7. Cap the vial.

8.3.8. Record all extraction information in the appropriate Extraction Logbook (see **Figures 1 and 2**).

8.4. The sample extract is now ready for filtration and concentration. These procedures are found in SOP No. 50.0054, Extract Filtration and Concentration.

8.5 After concentration the extract may require cleanup. The following is a list of Cleanup Procedures:

8.5.1. **Sulfur cleanup** is mandatory for all Pesticide/PCB sample extracts containing sulfur. All QC samples including blanks, lab control samples, matrix spikes and duplicate matrix spikes must be subjected to the same cleanup as the field samples. Sulfur cleanup will be performed using activated copper powder. Refer to SOP No. 50.0030 Method 3660B Sulfur Cleanup, for details on activiting copper and using it in a sample extract.

8.5.2. **Acid cleanup (PCB extract only)** is mandatory for all PCB sample extracts. Refer to SOP No. 50.0031 Method 3665A Sulfuric Acid Cleanup, for details on using the acid cleanup in a PCB sample extract.

8.5.3. **GPC cleanup** is useful for both Pesticide and semivolatile samples. Refer to SOP No. 50.0032 Method 3640A GPC Cleanup, for details on the procedure for cleanup and quality control criteria for GPC.

8.5.4. Other cleanup methods may be found in SOP numbers 50.0033(Florisil) and 50.0034 (Silica Gel).

8.6 The extracts are transferred to the Semivolatile lab and documented in the appropriate Extract Transfer Log. The extracts are stored in the refrigerator at 4°C until analysis.

8.7 Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

8.7.1. Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of by the Sample Custodian or his/her designee.

8.7.2. Sample Extracts – All sample extracts are kept for at least 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of by the GC or GC/MS labs.

9. Data Reduction and Calculations

Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{\text{(Concentration of ampule)(amount used)}}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. To trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and the manufacturer's reference number is documented in the standard preparation log book.

10.1. Method Blank – A method blank is a weight of a clean reference matrix (granular sodium sulfate) that is carried through the entire analytical procedure.

10.1.1. Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2. Procedure for Method Blank:

- The Method Blank is prepared in identical fashion to the associated samples.
- An aliquot of the surrogate standard prepared in **section 8.1.2** is added to the Method Blank.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a sequential number for every batch of twenty samples or less.

10.2. Lab Control Sample (LCS) – A Lab Control Sample is a weight of a clean reference matrix (granular sodium sulfate) that is spiked with all appropriate target analytes and surrogate spikes, and carried through the entire analytical procedure.

10.2.1. Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.2.2. Procedure for LCS:

The LCS is prepared in identical fashion as the associated samples; and in addition:

- An aliquot of the surrogate standard prepared in **section 8.1.2** and the lab control standard prepared in **section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the same numerical value as the corresponding method blank.
- The LCS is analyzed and the results are calculated for the recovery of all spiked analytes in the LCS.

10.3. Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to

evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.3.1. Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.3.2. Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition:

- An aliquot of the surrogate standard prepared in **section 8.1.2** and the lab control standard prepared in **section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.
- The duplicate matrix spikes are analyzed and the results calculated for the recovery of the spiked analytes in the duplicate matrix spike.

11. Data Validation and Reporting

11.1. Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of, but are not limited to, extraction/preparation logbook entries, balance calibration logbooks, weights for soil samples, volumes and lot numbers of solvent used. The Quality Control Officer will perform periodic and unscheduled reviews.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1. Method Blank Analysis:

All samples that are prepared with a non-compliant Method Blank will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The

analysis laboratory will inform the Organic Preparatory Laboratory when method blank contamination has occurred. A request for re-extraction will be filled out (**Figure 3**).

12.2. Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.3. LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.4. Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan; Mitkem Laboratories, A Division of Spectrum Analytical Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

Attachments:

Attachment 1: Pesticide LCS/MS List
Attachment 2: Sonicator Tuning instructions

Figure 1: Semivolatile Extraction Log
Figure 2: Pesticides/PCB Extraction Log
Figure 3: Re-extraction Request
Figure 4: Sonicator Tuning Log

Attachment 1: Pesticide LCS/MS List

Analyte	Spike conc in ug/mL
4,4´-DDD	0.4
4,4´-DDE	0.4
4,4´-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2

Attachment 2
Sonicator Tuning instructions

The User I/O may be used to remotely control the system. If this is the case, you must design in whatever safety precautions are appropriate to your User I/O circuit design to prevent unexpected start-up, which can cause personal injury and can cause equipment damage.

3.8 Ultrasonic Test

The Test button on the front panel of the Model 500 is used to verify that the unit is functioning (providing ultrasonic energy to the converter and horn). You can also run another test on the system for your particular experiment as described later. Before testing the Model 500, always make sure that the horn is not touching anything. The system will perform several self-tests when it is first turned on.

Step	Do this...	To obtain this result
1	Set up the Model 500 following the instructions in this manual. If no horn is currently installed, mount a 1/2" disruptor horn to the converter.	Prepare the Model 500 to operate, if it was not previously assembled.
2	After you have connected the converter/horn to the converter cable, verified all other connections are as desired: Turn the unit on, and observe the self-test displays.	Verify that the system passes all its self-tests, observing that there are no error messages on the front panel display. The Model 500 advances to the ready mode and shows the normal "Ready" display.
3	Adjust the amplitude control to approximately 50% (observe the value on the front panel display).	Ensures that ultrasonic energy will be at some mid-range value, and will not cause damage if you were using a microtip (must be less than 70%).
4	Verify that the horn is not touching anything. Press the test button on the front panel. Observe the front panel display.	Verifies the ultrasonic output of the system. You may hear a soft, high-pitched sound. The bargraph display will show some output value. The test will run for 2 seconds, then stop.
5	If the system showed readings on the display during the test, you may either proceed with your experiments or turn the unit off.	Verification that the Model 500 Dismembrator is operating and is ready to be set up for your experiment.

Sonic Dismembrator

Fisher Scientific

Model 500

F. TUNING INSTRUCTIONS - (for standard probes, Q-horns and cuphorns)

To assure optimum operation tune the generator in accordance to the following procedure each time a new probe is changed.

The probe or microtip should not be immersed in the liquid or come in contact with the work surface when tuning.

When operating with liquids at extreme temperatures, immerse the probe in the liquid for a few minutes, remove from the liquid then tune.

1. Turn OUTPUT CONTROL knob counter-clockwise to zero.

2. Press POWER SWITCH to ON (up) position. The switch will illuminate.

3. When the prompt [for tuning procedure refer to manual] appears, press TUNE key. Screen will read:
   [TUNING - - - PROBE ACTIVE].

4. Turn the Output Control Knob towards setting 3.
   a) Note the position of the Bar Graph on the LCD Display Screen. Do NOT exceed 70%.
   b) Rotate the Tuning Knob clockwise or counterclockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.

5. Turn Output Control Knob towards setting 6.
   a) Again, note the position of the Bar Graph and do not exceed 70%.
   b) Rotate the tuning knob till you obtain a meter reading of 20% or below.

6. Repeat step 5 on power setting 10. Minimize the reading one last time to 20% or less.

7. Press the TUNE key to display prompt for programmed or continuous operation.

YOUR SONIC DISMEMBRATOR IS NOW TUNED

SPECIAL NOTE FOR TUNING MICROTIP™ PROBES AND OTHER HORNS

MICROTIPS

The above procedure must be followed with the exception that the OUTPUT CONTROL should NEVER exceed the MICROTIP LIMIT (5). Tuning at the MICROTIP LIMIT should be done as quickly as possible. Prolonged operation in air at the limit can cause MICROTIP failure.

CUPHORNS

Drain filled cups down to outlet fitting level, tune, and refill.

Figure 1

Semivolatile Extraction Log

MITKEM LABORATORIES **ORGANIC PREP - SAMPLE PREPARATION: SEMIVOLATILES**

Date:	Analysis:	Method #	Aq: 3510C (SepF) 3520C (Liq/Liq) Other: Soil: 3550B (Sonic) 3540C (Soxhlet) _____	Matrix Aqueous Soil Wipe Oil Other:_____	Sodium Sulfate Lot #:

Batch ID	LCS ID	Analyst	Spiked By	Witness	MeCl Lot#	Acetone Lot#	H_2SO_4 Lot#	NaOH Lot#	Time/Date Started: Time/Date Ended:

Lab ID	Bottle #	Sample Wt (g) / Vol (mL)	Initial pH	Surrogate Spike Added(uL)	Matrix Spike Added(uL)	H2SO4 pH<2	NaOH pH>11	Emulsion	KD/RV Prior to GPC or Fractionation Date / Analyst	GPC or Fractionation Date / Analyst	Final Concentration Date / Analyst	Final Conc. Volume (mL)	Date Extract Trans.

Comments: _____

Surrogate Std. ID: _____

Matrix Spike/LCS Std. ID: _____

Sonicator Tuned: Yes / No

Soxhet Cycle/Hour:_____

Drip Rate_____

Water Bath Temp:_____

Logbook ID: 50.0147-01/10

Reviewed By:_____

Figure 2

Pest/PCB Extraction Log

MITKEM LABORATORIES ORGANIC PREP-SAMPLE PREPARATION:PEST/PCB

Date:	Prep Method	Aq: 3510C (SepF) 3520C (Liq/Liq) Soil: 3550B (Sonc) 3540C (Soxhlet) 3570(MSE) Other:_____	Matrix: Aqueous Soil Wipe Oil Other:_____	BATCH ID:	Date/Time Started:

Analyst	Spiked By	Witness	Solvent: Lot #	GPC Batch Number	Florisil Lot #	Analysis:	Date/Time Ended:

	Lab ID	Bottle #	Sample Wt (g) / Vol (ml)	Initial pH	Emulsion?	Initial CONC Date/ Analyst TV/ KD	Extract volume (ml)	GPC Cleanup Date / Analyst	FLORISIL Cleanup Date / Analyst	ACID (PCB only) Cleanup Date / Analyst	SULFUR (Copper) Cleanup Date / Analyst	Final CONC Date/ Analyst TV/ KD	Final extract volume (ml)	Extract Transfer Date /By

Comments:

PEST Matrix Spike Std ID:	Vol. Spiked:		
PCB Matrix Spike Std ID:	Vol. Spiked:		
PEST Surrogate Spike Std ID:	Vol. Spiked:	Sodium Sulfate lot#	
PCB Surrogate Spike Std ID:	Vol. Spiked:	Hexane lot #	L/L Drip Rate:
PEST LCS Spike Std ID:	Vol. Spiked:	Sulfuric Acid lot#	Soxhlet Cycle/Hour:
PCB LCS Spike Std ID:	Vol. Spiked:	Sulfur cleanup Copper lot #	Sonicator Tuned? Yes/No

Figure 3
Re-extraction Request

Mitkem Laboratories, A Division of Spectrum Analytical, Inc.
RE-EXTRACTION LOGBOOK
ORGANICS

REQUESTED BY:
DATE:

Mitkem Sample ID	Sample Matrix	Analysis	MB originally associated with sample	Reasons for Re-extraction

PREP LAB APPROVAL:

DATE:

Logbook ID 50.0190-06/08

Reviewed By:_____

Figure 4:
Sonicator Tuning Log

Mitkem Laboratories Organic Prep Sonicator Horn Tuning Logbook

See individual Manufacturer Manuals* for instructions on tuning, and passing criteria.

Date	Horn ID	Pass	Fail	Comments	Analyst

Logbook ID: 50.0014-09/09 **Reviewed by:** _____

*POSTED BESIDE HOOD

MITKEM LABORATORIES
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Organic Extract Filtration and Concentration Techniques

Rev 2

	Signature	Date
QA Director:		2/2/10
Lab Director:		2/4/10
Effective Date:	02/11/10	

MITKEM LABORATORIES
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Organic Extract Filtration and Concentration Techniques
Rev. 2

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the filtration and concentration of organic sample extracts that were prepared using EPA water and SW-846 solid hazardous waste methods, as well as OLC3.2, OLM4.3 and the current SOM version procedures.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

The sample extract is dried through granular anhydrous sodium sulfate and is made ready for cleanup and/or analysis following concentration using Kuderna-Danish (KD) apparatus or Caliper TurboVap apparatus and nitrogen blow down.

4. Sample Preservation, Containers, Handling, and Storage

Not Applicable

5. Interferences and Potential Problems

5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation and concentration; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results for the analytes of interest. See Mitkem SOP Nos. 50.0030 through 50.0034 for cleanup methods.

5.3 Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation and concentration steps to minimize interferences from these compounds.

6. Equipment and Apparatus

Equipment and apparatus used in this method include:

6.1 Teflon boiling chips.

6.2 Kuderna-Danish (KD) apparatus with a 10 or 15ml receiver tube.

6.3 80mm Glass funnel.

6.4 Glass wool.

6.5 Three ball Snyder column.

6.6 Water bath, capable of maintaining 60 °C to 90°C.

6.7 Boiling chips, carbon.

6.8 Nitrogen blow-down apparatus, N-EVAP Model No. 111.

6.9 Receiving vial with Teflon septa – 2ml.

6.10 15ml storage vials with screw caps.

6.11 Caliper TurboVap apparatus with 200mL collection tube

6.12 Aluminum Foil (Industrial Grade)

6.13 Glass Pipettes

6.14 20X150mm Disposable Culture Tubes

7. Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the

accuracy of the test. The following chemicals including solvent, standards and gasses are extensively used in the lab:

7.1. Methylene chloride: pesticide quality or equivalent, to be used for glassware rinsing and sample extraction

7.2. Hexane: pesticide quality or equivalent, to be used for solvent exchange of samples.

7.3. Anhydrous sodium sulfate, granular for drying the sample extract.

7.4. Acetone: pesticide quality or equivalent, to be used for glassware rinsing.

8. Procedure

8.1 Sample Extract Filtration:

All sample extracts are dried through anhydrous sodium sulfate prior to further sample concentration.

8.1.1. A drying funnel is first cleaned with methylene chloride to remove any contaminants. The drying funnel is then prepared by plugging an 80mm diameter filtering funnel with glass wool and adding approximately 20 grams of anhydrous granular sodium sulfate.

8.1.2. Sample extracts are dried through the drying funnels and collected directly into the KD concentrators or Caliper TurboVap collection tubes depending on which method of concentration is going to be used. Rinse the empty boiling flask and drying funnel with methylene chloride after the initial transfer to ensure the qualitative transfer of the extract.

8.1.3. The KD concentrator apparatus consist of a 500ml KD flask and a 10 or 15ml concentrator tube. The concentrator tube is attached to the flask by placing it on and then giving a gentle twist. The concentrator tube should be on snug. Place a blue Keck clamp to ensure the tube will not fall off. Continue with **Section 8.2.1**.

8.1.4. The Caliper TurboVap 200mL collection tubes are placed underneath the drying funnel and the extract is collected directly into it. Continue with **Section 8.2.2**.

8.2. Sample Extract Concentration:

8.2.1. Procedure for concentration of sample extracts using **KD** apparatus without further Gel Permeation Chromatography (GPC) cleanup.

8.2.1.1. Add 2 boiling chips to the KD flask containing the extraction fluid.

8.2.1.2. Attach a three-ball Snyder column to the KD flask and submerge the evaporator into the hot water bath set at medium heat (recommended temperatures are: 60-70 °C for SV (or DRO) and as high as 80-90°C for Pest/PCB during and after hexane exchange) with the tip of the KD concentrator tube in the water.

8.2.1.2.1. Concentrate the **SV (or TPH/DRO)** extract down to about 1ml.

8.2.1.2.2. Concentrate the **Pest/PCB** extract down to less than 5ml and then add approximately 20ml of hexane through the top of the Snyder column to exchange the solvent. Continue to concentrate the extract to less than 5ml. REPEAT.

Caution: It is important that the extract in the KD apparatus not be allowed to go dry to minimize volatilization of the more volatile target compounds.

8.2.1.3. Remove the evaporator from the water bath. Let the evaporator cool for at least 10 minutes, allowing the solvent in the Snyder column to drain back into the KD concentrator tube. Rinse the KD flask with a small volume of methylene chloride.

8.2.1.4. Remove the three-ball Snyder column and the KD flask from the concentrator tube. Transfer the extract into a 20x150mm disposable culture tube. Rinse the lower joint of the KD flask and the concentrator tube with another small amount of methylene chloride to ensure a full transfer of the extract.

8.2.1.5. Place the culture tube into the nitrogen blow down bath, set at 30-35°C (recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8.

8.2.1.6. Start a low flow stream of nitrogen across the surface of the extraction solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulate inside the tube.

Caution: it is important that the extract in the culture tube not be allowed to go dry to minimize loss of the more volatile target compounds.

8.2.1.7. Concentrate the **SV (or TPH/DRO)** extract to between 0.5 and 0.8ml. Pull the extract up with a 1.0ml syringe. Rinse the collection tube with a small volume of methylene chloride and pull up the rinsate filling the syringe to the 1.0ml mark. Transfer the 1.0ml extract to a 2ml auto-sampler vial. Mark the meniscus with a permanent marker.

8.2.1.8. Concentrate the **Pest/PCB** extract to 5ml. Transfer the extract to a 15ml vial marked at the 10ml mark. Rinse the concentrator tube with 1ml of hexane

and add to vial. Rinse the concentrator tube once more. Bring the final extract volume up to 10ml with hexane. Mark the meniscus with a permanent marker.

8.2.2. Procedure for concentration of sample extracts using TurboVap (TV) apparatus without Gel Permeation Chromatography (GPC) cleanup:

8.2.2.1. Place the 200mL TV collection tube into the TurboVap. Set the nitrogen to 10 psi, and the temperature to 60 °C (recommended).

8.2.2.1.1. Concentrate the **Semivolatile** extracts to 0.5 – 0.8mL. Allow the collection tube to stand without nitrogen flow to cool. Using a 1.0 ml syringe pull up the extract and rinse the interior of the collection tube with the extract. Pull up the extract again with the 1.0 ml syringe. Rinse the collection tube with additional methylene chloride and pull up in the syringe. If needed, add additional methylene chloride to bring the extract to 1.0 ml. Transfer the extract to a 2 ml crimp top autosampler vial. Mark the meniscus with a permanent marker.

8.2.2.1.2. Concentrate the **Pesticide/PCB extract** to about 4ml and then add 20ml of hexane. Raise the temperature to 80 °C (recommended), if necessary, to complete the concentration. Concentrate to about 4 ml again, and add 20 ml of hexane. Continue to concentrate the extract as in **Section 8.2.1.5.2.**

Caution: it is important that the extract in the collection tube not be allowed to go dry to minimize volatilization of the more volatile target compounds.

8.2.2.2. The extracts are transferred to the GC or GC/MS lab and documented in the appropriate Extract Transfer log. The extracts are stored in the appropriate refrigerator, at 4°C ± 2 °C until analysis.

8.2.3. Procedure for concentrating sample extracts using the **KD** apparatus with further Gel Permeation Chromatography (GPC) cleanup:

8.2.3.1. Add 2 boiling chips to the KD flask containing the extraction fluid.

8.2.3.2. Attach a three-ball Snyder column to the flask and submerge the evaporator into the water bath set at medium heat (recommended temperatures are: 60-70 °C for SV (or DRO) and as high as 80-90°C for Pest/PCB during and after hexane exchange) with the tip of the KD concentrator tube in the water.

8.2.3.3. Concentrate the extract down to less than 5ml and remove the evaporator from the water bath.

8.2.3.4. Let the evaporator cool for at least 10 minutes, and allow the solvent in the Snyder column to drain back into the concentrator tube. Rinse the KD flask with a small volume of methylene chloride to ensure a full transfer of the extract.

8.2.3.5. Remove the three-ball Snyder column and the KD flask from the concentrator tube. Transfer the extract into a disposable culture tube and place the culture tube into the nitrogen blow down bath, with the temperature set at 30-35°C(recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8. Rinse the lower joint of the KD flask and the concentrator tube with a small amount of methylene chloride to ensure a full transfer of the extract.

8.2.3.6. Start a low flow stream of nitrogen across the surface of the extract solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulates inside the tube. Concentrate the samples to 2ml. This is to reduce the concentration of acetone in the Pre-GPC soil extract.

8.2.3.7. Transfer the 2ml extract into a 15ml vial with a line at the 10ml mark. Rinse the concentrator tube with 2ml of methylene chloride and combine the rinsate into the vial. Rinse the concentrator tube one more time with 2ml of methylene chloride and pour into the vial. Bring up to the 10ml mark with methylene chloride.

8.2.3.8. The extract is now ready for GPC cleanup. Refer to SOP 50.0032 for how to perform GPC.

8.2.4. Procedure for concentrating sample extracts using the **TV** apparatus with further Gel Permeation Chromatography (GPC) cleanup:

8.2.4.1. Place the 200mL TV collection tube into the TurboVap. Set the nitrogen to 10 psi, and the temperature to 60 °C (recommended).

8.2.4.2. Concentrate the extract down to about 2-3 ml and remove the collection tube from the nitrogen blow down bath. Allow to cool without nitrogen flow.

8.2.4.3. Pipet or carefully pour the extract into a 15mL vial marked with a line at the 10ml mark. Rinse the concentrator tube with 2mL of methylene chloride and pour into the vial. Rinse the concentrator tube one more time with 2mL of methylene chloride and pour into the vial. Bring up to the 10mL mark with methylene chloride.

8.2.4.4. The extract is now ready for Gel Permeation Chromatography (GPC) cleanup. Refer to SOP 50.0032 for how to perform GPC Method SW3640.

8.2.5. Post GPC the extracts require additional concentration steps.

8.2.5.1. For **Semivolatile** extracts, 5ml of the original 10 ml extract was used for cleanup and the final extract volume must be 0.5ml in methylene chloride.

8.2.5.1.1. Semivolatile extracts (TPH/DRO samples do not undergo GPC Cleanup) are re-concentrated using the KD until approximately 4ml.

8.2.5.1.2. The extract is then transferred to 20x150mm disposable culture tubes and placed into the nitrogen blow down bath, set at 30-35°C(recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8.

8.2.5.1.3. Start a low flow stream of nitrogen across the surface of the extraction solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulate inside the tube.

Caution: it is important that the extract in the culture tube not be allowed to go dry to minimize loss of the more volatile target compounds.

8.2.5.1.4. Concentrate the **SV** extract to a final volume of 0.5ml. Pull the extract up with a 1.0ml syringe. Rinse the collection tube with a small volume of methylene chloride and pull up the rinsate filling the syringe to the 0.5ml mark. Transfer the extract to a 2ml auto-sampler vial. Mark the meniscus with a permanent marker.

8.2.5.2. For **Pesticide/PCB** extracts, 5ml of the original 10 ml extract was diluted to a 10ml final volume using methylene chloride, and was used for GPC cleanup. The final extract should be solvent exchanged to hexane and concentrated to 5ml.

8.2.5.2.1. The post GPC extract is transferred to a TV apparatus with the temperature set to 60 °C(recommended). Concentrate to about 4 ml. Raise the temperature to 80 °C(recommended), if necessary, to complete the concentration. Add 20ml of hexane to exchange the solvent. REPEAT. Continue to concentrate the extract until it has reached 2-3ml. Transfer the extract to a 15mL vial marked at the 5mL mark. Rinse the collection tube with hexane several times to ensure a full transfer. The final volume must be 5ml. Mark the meniscus with a permanent marker.

8.2.5.2.2. The extract is ready for florisil cleanup. Refer to SOP 50.0033 for detailed instructions on florisil cleanup, Method SW3620.

8.2.5.2.3. Other cleanup methods may be employed if needed. Possible procedures may be: Silica Gel Cleanup, Method SW3630 (SOP

50.0034), Acid Cleanup, Method SW3665 (SOP 50.0031) or Sulfur Cleanup, Method SW3660 (SOP 50.0030).

8.2.6. Excess volume of extracts are stored in screw cap vials and labelled with color coded lab tape. The color of the tape records the extract holding time until disposal. See chart in laboratory.

8.3. Sample and Extract Disposal:

All sample extracts are disposed of in accordance with applicable OSHA and state regulations. All sample extracts must be protected from light and stored at 4°C ± 2°C.

8.3.1. Sample Extracts – All sample extracts are kept for 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of.

8.3.2. EPA CLP/SOM sample extracts- All sample extracts are kept until 365 days after delivery of a reconciled, complete data package.

9. Data Reduction and Calculations

Not Applicable

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. In order to trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and contains the manufacturer's reference number given in the standard preparation logbook.

10.1. Method Blank – A method blank is a liter of organic free reagent water that is carried through the entire analytical procedure. It is used to determine the level of contamination associated with the analytical processing and analysis of samples.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank

- The Method Blank is prepared in identical fashion as the associated samples.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a numerical value, which increases with every batch of twenty samples or less.

10.2. Surrogate – Surrogate standards are added to all samples including the Method Blank, Lab Control Sample and matrix spikes to assess the efficiency of the sample preparation and analysis procedures.

10.3. Lab Control Sample (LCS) – A Lab Control Sample is a liter of organic free reagent water that is spiked with all target analytes and the surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1. Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.3.2. Procedure for LCS:

An LCS is prepared in identical fashion as the associated samples; in addition:

- An aliquot of surrogate solution prepared in **Section 8.1.2** and lab control spike prepared in **Section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the corresponding numerical value as the associated method blank.

10.4. Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix. For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.4.1. Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Once every 20 samples, or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.4.2. Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples, in addition:

- An aliquot of surrogate solution prepared in **Section 8.1.2** and lab control spike prepared in **Section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.

11. Data Validation and Reporting

11.1 Data generated in the organic preparation laboratory will be reviewed by the supervisor. The Quality Control Officer will perform periodic and unscheduled reviews. These data consist of the final volume of sample extracts, the volume and lot number of solvents used, and extract transfer dates.

11.2 Reporting of the data will include review by the Organic Preparation Laboratory Supervisor of the data listed in **Section 11.1**, time of extraction, sampling handling procedures, and extract handling procedures.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1 Method Blank Analysis:

All samples associated with a non-compliant Method Blank are re-extracted and reanalyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when method blanks have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out. The re-extracted samples will be labeled with the suffix RE.

12.2 Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The analysis laboratory will inform the Organic

Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out.
The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects.

12.3 LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. Any sample(s) that is/are associated with a non-compliant LCS will require re-extraction and re-analysis. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out .
The re-extracted samples will be labeled with the suffix RE.

12.4 Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, IV, or On-line Revisions of 3500 and 3600 series for Organic Extraction, Sample Preparation and Cleanup.

USEPA Statement of Work, Current OLC, OLM and SOM Methods.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

For

THE DETERMINATION OF

PCB HOMOLOGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

/SINGLE ION MONITORING(SIM) USING MODIFIED EPA METHOD 680

SOP No. 60.0054 rev. 0

	Signature	Date
QA Director:		2/5/08
Lab Director:		2/7/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

THE DETERMINATION OF

PCB HOMOLOGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

/SINGLE ION MONITORING(SIM) USING MODIFIED EPA METHOD 680

1. Scope and Application

This method provides procedures for mass spectrometric determination of polychlorinated biphenyls (PCBs) in sediment. This method is applicable to samples containing PCBs as single congeners. PCBs are identified and measured as isomer groups or homologs *(i.e.,* by level of chlorination). The existence of 209 possible PCB congeners makes the listing of the Chemical Abstracts Service Registry Number (CASRN) for each potential method analyte impractical. Because PCBs are identified and measured as isomer groups, the non-specific CASRN for each level of chlorination is used to describe method analytes.

Analyte(s)	Formula	CASRN
Monochlorobiphenyls	$C_{12}H_9Cl$	27323-18-8
Dichlorobiphenyls	$C_{12}H_8Cl_2$	25512-42-9
Trichlorobiphenyls	$C_{12}H_7Cl_3$	25323-68-6
Tetrachlorobiphenyls	$C_{12}H_6Cl_4$	26914-33-0
Pentachlorobiphenyls	$C_{12}H_5Cl_5$	25429-29-2
Hexachlorobiphenyls	$C_{12}H_4Cl_6$	26601-64-9
Heptachlorobiphenyls	$C_{12}H_3Cl_7$	28655-71-2
Octachlorobiphenyls	$C_{12}H_2Cl_8$	31472-83-0
Nonachlorobiphenyls	$C_{12}HCl_9$	53742-07-7
Decachlorobiphenyls	$C_{12}Cl_{10}$	2051-24-3

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors** review the logbooks and data generated from this procedure and approve all reported results. The **Project Manager** evaluates laboratory reports for reasonableness of the results and signs the reports. The **QA Director** reviews quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure

In general, samples are extracted with a pesticide-grade solvent. The extracts are further processed by concentrating or diluting, depending on the PCB concentration, and carried through a series of cleanup techniques. The sample is then analyzed by injecting the extract onto a gas chromatographic system and the PCBs detected by a mass spectrometer.

This SOP provides detailed instructions for gas chromatographic/mass spectrometer conditions, calibration, and analysis of PCBs by gas chromatography/mass spectrometry (GC/MS). Sediment extraction will be performed using SW-846 Method 3545 Pressurized Fluid Extraction or Method 3550B Sonication , using 1:1 methylene chloride and acetone solvents following Mitkem SOP Number 50.0057 or 50.0052. If aqueous QC samples are collected (field blanks, rinsates, etc.), they will be prepared for analysis using either the continuous liquid-liquid extraction technique SW-846 Method 3520C, or separatory funnel technique SW-846 Method 3510C, using the current revision of Mitkem SOP 50.0050 or 50.0051.

Sample extract components are separated with capillary column gas chromatography (GC) and identified and measured with low resolution, electron ionization mass spectrometry (MS). An interfaced data system (DS) to control data acquisition and to store, retrieve, and manipulate mass spectral data is essential. Selected-ion-monitoring (SIM) data are to be acquired. In SIM mode the mass spectrometer scans only certain atomic masses, those characteristic for the compounds of interest. This divides the MS sensitivity over a much smaller mass range, which results in enhanced sensitivity. At the laboratory's option, extracts may be pre-screened, by a suitable procedure, to help determine the appropriate initial dilution or to avoid overloading the analytical system.

Mitkem will use an Agilent (formerly Hewlett Packard) Model 6890 or 7890 GC interfaced to a Mass Selective Detector (MSD). MSD Models 5972, 5973 or 5975 have been used successfully. This instrument is a quadrupole mass spectrometer, which is specified for use in most other EPA analytical methods.

Two surrogate compounds are added to each sample before sample preparation; these compounds are the carbon 13 (C-13) analogs of Tetrachlorobiphenyl, 3,3',4,4' and Octachlorobiphenyl, 2,2',3,3',4,4',5,5' . One internal standard, the carbon 13 (C-13) analog of Tetrachlorobiphenyl, 2,2',5,5' is added to each sample extract before GC/MS analysis and are used to calibrate MS response. Each concentration measurement is based on an integrated ion abundance of one characteristic ion for each homolog.

PCBs are identified and measured as isomer groups or homologs (*i.*e., by level of chlorination). A concentration is measured for each PCB isomer group total; total PCB concentration in each sample extract is obtained by summing isomer group concentrations. Throughout this method, individual PCBs are described with the number assigned by Ballschmiter and Zell (This number is also used to describe PCB congeners in catalogs produced by Ultra Scientific, Hope, RI.).

4. Sample Preservation, Containers, Handling, and Storage

4.1 Samples are collected by clients and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients provide their own containers. Water samples are collected in 1-liter amber glass bottles with no preservation added to the samples. Solid samples are collected in 8-ounce amber glass containers with no preservation needed. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may be required for the analysis of laboratory QC samples.

4.2 As an option for sediment samples, the EPA has approved storage of samples in a freezer as a means of extending the holding time. The holding time "clock" will not advance while samples are frozen, but only advances while samples are not frozen. The date that samples are initially frozen, withdrawn from the freezer for analysis, returned to the freezer, and any subsequent removal and re-freezing must be recorded.

4.3 All sample extracts are stored at 4°C ± 2°C until analyzed.

4.4 Sample extract holding time for PCB Homolog analysis by the method is 40 days from date of extraction to date of analysis. The holding time for sample extraction is covered in the corresponding extraction SOPs.

5. Interferences and Potential Problems

Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing equipment. Method blanks are analyzed routinely to demonstrate that these materials are free of interferences under the analytical conditions used for samples.

To minimize interferences, glassware (including sample bottles) should be meticulously cleaned. As soon as possible after use, rinse glassware with the last solvent used. Then wash with detergent in hot water and rinse with tap water followed by distilled water. Drain dry. Heating in a muffle furnace at 450 °C for a few hours may be used as a further cleaning technique, but does not have to be performed provided that method blanks demonstrate glassware cleanliness. After cooling, store glassware inverted or covered with aluminum foil. Before using, rinse each piece with an appropriate solvent. Volumetric glassware must not be heated in a muffle furnace.

6. Equipment and Apparatus

6.1 The GC must be capable of temperature programming and be equipped with all required accessories, such as syringes, gases, and a capillary column. The GC injection port must be designed for capillary columns. Splitting injections is not recommended.

6.1.1 SIM mass spectral data are obtained with electron ionization at a nominal electron energy of 70 eV. To ensure sufficient precision of mass spectral data, the required MS scan rate must allow acquisition of at least five full-range mass spectra or five data points for each monitored ion while a sample component elutes from the GC. The MS must produce a selected ion mass spectrum for each calibration congener meeting all criteria for the quant and confirmation isotope ratios listed in Table 9.

6.1.2 An interfaced data system (DS) is required to acquire, store, reduce, and output mass spectral data. The DS must be capable of searching a data file for specific ions and plotting ion abundances versus time or spectrum number to produce selected ion current profiles (SICPs) and extracted ion current profiles (EICPs). Also required is the capability to obtain chromatographic peak areas between specified times or spectrum numbers in SICPs or EICPs. Total data acquisition time per cycle should be $\geq$ 0.5 s and must not exceed 1.5s.

6.1.3 SIM – For SIM data acquisition, the DS must be equipped with software capable of acquiring data for multiple groups of ions, and the DS must allow automated and rapid changes of the set of ions being monitored. The SIM program must be capable of acquiring data for five groups (or mass ranges) each consisting of $\leq$ 27 ions each. The times spent monitoring ions during sample analyses must be the same as the times used when calibration solutions were analyzed.

6.2 GC COLUMN – A 60 m × 0.25 mm ID fused silica capillary column coated with a 0.25 µm film, Durabond-5, Agilent Technologies is required. Operating conditions known to produce acceptable results with this column are shown in **Table 1**. Separation of PCB calibration congeners with a DB-5 column and those operating conditions is shown in **Figure 1**. **Figure 2** shows a chromatogram of the PCB Window Defining Mixture used to determine retention time windows for the five ion groups for SIM data acquisitions.

6.3 Volumetric flasks -- 2-mL, 5-mL, 10-mL, 25-mL, and 50-mL with ground glass stoppers.

6.4 Microsyringes -- various standard sizes 9.

6.5 Analytical Balance -- capable of accurately weighing to 0.0001 g.

7. Reagents and Standards

7.1 SOLVENTS -- High purity, distilled-in-glass hexane, methylene chloride and acetone. For precise injections with splitless injectors and capillary columns, all samples and standards should be contained in the same solvent. Effects of minor variations in solvent composition *(i.e.,* small percentage of another solvent remaining in hexane extracts) are minimized with the use of internal standards. (External standard calibration is not acceptable.)

7.2 SODIUM SULFATE - ACS, granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray, or purchase pre-baked material.

7.3 SURROGATE COMPOUNDS: (Cambridge Isotopes at 40 µg/mL) Carbon-13 analogs of Tetrachlorobiphenyl, 3,3',4,4' and Octachlorobiphenyl, 2,2',3,3',4,4',5,5' are added to every sample before extraction and are included in every calibration/performance check solution and quality control samples.

7.4 PCB CONCENTRATION CALIBRATION CONGENERS - The nine individual PCB congeners listed on **Table 2** are used as concentration calibration compounds for PCB determinations. One isomer at each level of chlorination is used as the concentration calibration standard for all other isomers at that level of chlorination, except that decachlorobiphenyl (Cl_{10}) is used for both Cl_9 and Cl_{10} isomer groups.

7.5 PCB RETENTION TIME CONGENERS FOR SIM DATA ACQUISITION -- Knowledge of the retention time of certain congeners is necessary to determine when to acquire data with each ion set. Two concentration calibration congeners also serve as retention time congeners; the first eluting Cl1 -PCB indicates the time when data acquisition must have been initiated for ion set #1, and the Cl10 -PCB indicates when all PCBs have eluted. A PCB Window Defining Mixture Standard (Ultra, catalog item CB-682MN or equivalent) is analyzed at a concentration of 2.5ug/mL for each PCB congener. The PCB Window Defining Mixture Standard contains the first and last eluting PCB congener for each Homolog group. The following four congeners are used from this standard to define the five retention time segments for the five Ion Set Groups: BZ#104, BZ#77, BZ#202, and BZ#189. (See Sect. 8.1.2 for Ion Set Segments).

7.6 PCB SOLUTIONS:

7.6.1 Primary Homolog Calibration Mix standard (Ultra Scientific, catalog item CB-681MN or equivalent) -- a stock solution containing all of the appropriate PCB congeners at concentrations of 50-250 µg/mL in hexane is obtained from Ultra Scientific. Place the solution in a clean glass vial with a Teflon-lined screw cap and store at 4 °C if solution is not to be used right away. The Solution is stable indefinitely if solvent evaporation is prevented.

CAUTION: Each time a vial containing small volumes of solutions is warmed to room temperature and opened, a small volume of solvent in the vial headspace evaporates, significantly affecting concentration. Solutions should be stored with the smallest possible volume of headspace, and opening vials should be minimized.

7.6.2 CAL FOR SIM DATA ACQUISITION -- One set of five solutions is needed for PCB determinations. Appropriate concentrations of SIM CALs are given on **Table 3 and 4**. The five solutions are prepared by diluting the primary Homolog Calibration Mix standard and the two surrogates together in hexane. An appropriate volume of IS solution is added to 1.0mL of each of the five level standards to provide Tetrachlorobiphenyl, 2,2',5,5' to achieve a concentration of 0.80 ng/µL (ppm). Calculate the concentration (two significant

figures if <100 and three significant figures if >100 ng/µL) of each component in each solution.

7.7 INTERNAL STANDARD (IS) SOLUTION–The carbon-13 analog of Tetrachlorobiphenyl, 2,2',5,5' at a concentration of 40 ng/µL (ppm). A stock standard of 40 ng/µL of the carbon-13 analog of Tetrachlorobiphenyl, 2,2',5,5' in iso-octane is purchased from Cambridge Isotope Laboratories.

7.8 INITIAL CALIBRATION VERIFICATION (ICV) – A standard at the same approximate concentration as the Medium CAL is used as the initial calibration verification to demonstrate the accuracy of the primary calibration standards. The ICV is ordered from Chem Service, or other suitable supplier, but must be different from the supplier of the primary calibration standards (second source).

8. Procedure

Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required intermittently throughout sample analyses as dictated by results of continuing calibration checks. After initial calibration is successfully performed, a continuing calibration check is required at the beginning and end of each 12-h period during which analyses are performed.

8.1 INITIAL CALIBRATION

8.1.1 The mass spectrometer is tuned using Agilent's autotune routine against the calibration gas (perfluorotributylamine - PFTBA), modified by maximizing response for ions 414 and 502.

The MS must produce a selected ion mass spectrum for each calibration congener meeting all criteria for the quant and confirmation isotope ratio listed in Table 9. If the ~~spectrum~~ spectra do not meet all criteria), the MS must be hardware tuned to meet all criteria before proceeding with calibration. Investigate source of problem, reinject the standard if data indicate a potential bad injection, evaluate instrument response to cal gas (perfluorotributylamine - PFTBA), adjust MS settings via hardware tune to improve performance. When instrument response to PFTBA is improved, re-inject the standard and evaluate results. Once the mass spectrometer passes the isotope criteria, all subsequent blanks and standards that are analyzed within the 12-hour shift must be analyzed using identical mass spectrometer instrument conditions.

8.1.2 SIM Calibration – Inject a 4-µL aliquot of the Medium CAL standard. Acquire at least five data points for each ion during elution of each GC peak. Total cycle time should be ≥ 0.5 s and <1.5 s.

CAUTION: When acquiring SIM data, GC operating conditions must be carefully reproduced for each analysis to provide reproducible retention times; if not, ions will not be monitored at the appropriate times. SIM data acquisition provides an increase in sensitivity from full mass range data acquisition by at least a factor of three for PCB determinations.

8.1.2.1 Data will be acquired with the five ion sets (≤ 27 ions each) shown on **Tables 6 and 7**. The time (scan number) for initiation of data acquisition with each ion set must be carefully determined from the retention times (scan numbers) of the retention time congeners.

8.1.2.2 SIM data acquisition with five ion sets. Begin data acquisition with Ion Set #1 before elution of PCB congener #1, the first eluting Cl1 -PCB. Stop acquisition with Ion Set #1 and begin acquisition with Ion Set #2 just (approximately 10 seconds) before elution of PCB congener #104, the first eluting Cl5 -PCB. Stop acquisition with Ion Set #2 and begin acquisition with Ion Set #3 just (approximately 10 s) after elution of PCB congener #77, the last eluting Cl4 -PCB. Stop acquisition with Ion Set #3 and begin acquisition with ion Set #4 just (approximately 10 s) before elution of PCB congener #202, the first eluting Cl8-PCB. Stop acquisition with Ion Set #4 and begin acquisition with Ion Set #5 just (approximately 10 s) after elution of PCB congener #189, the last eluting Cl7-PCB, stop acquisition of Ion Set #5 after Cl10-PCB elution.

8.1.2.3 Evaluation of Calibration, Performance Criteria:

- GC separation -- Baseline separation of PCB congener #87 from congeners #154 and #77, which may coelute.
- MS sensitivity -- Signal/noise ratio of ≥ 5 for m/z 499 of PCB congener #209, Cl10 -PCB.
- MS calibration -- Abundance of ≥70% and ≤ 95% of m/z 500 relative to m/z 498 for congener #209, Cl10 -PCB.

If all performance criteria are met, analyze one 2-uL aliquot of each of the other four CAL standards to create the Initial Calibration.

8.2 CONTINUING CALIBRATION CHECK

8.2.1 With the following procedures, verify initial calibration at the beginning and after the end of each 12-h period during which analyses are to be performed using a continuing calibration verification check. If time remains in the 12-hour time period which includes the Initial Calibration, samples may be analyzed using the mid-point ICAL standard as the continuing calibration verification. Note—the continuing calibration verification that ends one 12 hour sequence can also serve as the calibration check at the beginning of the next 12 hour sequence, provided all performance criteria are met.

8.2.2 Calibrate and tune the MS with standards and procedures prescribed by the manufacturer.

8.2.3 SIM Analyte Retention Time Reproducibility -- Demonstrate and document acceptable (Sect. 9.4) reproducibility of absolute retention times of appropriate PCB retention time congeners by analysis of the PCB Window defining mixture in full scan mode. Analyze a 4-µL aliquot of the PCB Window solution and ensure acceptable MS calibration and performance (**Table 9**), following procedures in Section 8.1.1 for each subsequent calibration level.

8.2.4 Inject a 4-µL aliquot of the Medium CAL and analyze with the same conditions used during Initial Calibration.

8.2.5 Demonstrate acceptable performance for criteria described in **Sect. 8.1.2.3**.

8.2.6 Determine that the area measured for m/z 303 for Tetrachlorobiphenyl, 2,2',5,5'-C13 has not decreased by more than 30% from the area measured in the most recent previous analysis of a calibration solution or by more than 50% from the mean area measured during initial calibration.

8.2.7 Response Factor Reproducibility – The percent difference (%D) between the measured RF for each analyte/surrogate compound (RF_c) and the mean value calculated (Sect. 9.3) during initial calibration can be calculated as follows:

$$\%D = \frac{RF_c - RF_m}{RF_m} \times 100\% \qquad (5)$$

For an acceptable Continuing Calibration Verification (CCV), the %D for each PCB congener must be within ±20%. If the %D exceeds ±20%, the CCC is still deemed acceptable if mean %D for the nine PCB congeners is within ±20% and the %D for any one of these PCB congeners does not exceed 30%. If not, remedial action must be taken; recalibration may be necessary. The %D for each congener will be reported on the continuing calibration summary (Form 7). Any %D greater than 20% will be identified by flagging with an "*".

8.2.8 Remedial actions must be taken if criteria are not met; possible remedies are:

- Check and adjust GC and/or MS operating conditions.
- Clean or replace injector liner; replace septum and/or gold seal; and replace graphite o-ring.
- Flush column with solvent according to manufacturers instructions.
- Break off a short portion (approximately 0.33 m) of the column; check column performance by analysis of performance check solution.
- Replace GC column; performance of all initial calibration procedures is then required.

- Clean up the ion source according to the procedures provided by the manufacturer.
- Adjust MS for greater or lesser resolution.
- Calibrate MS mass scale.
- Prepare and analyze new concentration calibration/performance check solution.
- Prepare new concentration calibration curve(s).

8.3 GC/MS ANALYSIS

8.3.1 Remove the blank or sample extract and internal standard spiking solution from storage and allow them to warm to ambient laboratory temperature, if necessary. Add an appropriate volume of the appropriate internal standard stock solution.

8.3.1.1 Internal standard concentration for SIM data acquisition = 20µL of 40 ng/µL solution (Tetrachlorobiphenyl, 2,2',5,5'-C13) is added to 1.0 mL of extract for a concentration of 0.80 ng/µL.

8.3.2 Inject a 2-µL aliquot of the blank/sample extract into the GC operated under conditions used to produce acceptable results during calibration.

8.3.3 Acquire mass spectral data with SIM conditions. Use the same data acquisition time and MS operating conditions previously used to determine response factors.

8.3.4 Examine data for saturated ions in mass spectra of target compounds, if saturation occurred, dilute and reanalyze the extract after the quantity of the internal standards is adjusted appropriately to make sure that the internal standard concentration in the extract after dilution is still 0.80 ng/µL. In addition, any individual PCB analyte amount that exceeds the high level calibration standard of the calibration curve will require dilution and re-analysis of the extract to place that analyte within the calibration range.

8.3.5 For the internal standard, determine that the area measured in the sample extract has not decreased by >30% from the area measured during the most recent previous analysis of a calibration solution or by >50% from the mean area measured during initial calibration. If either criterion is not met, the data must be evaluated and corrective actions taken. The internal standard area may indicate a potential instrument problem, or matrix interference. Possible corrective actions include:

(1) Check the recovery of surrogate, and if the recovery is within the QC limits, the low area count is possibly due to sample matrix effect or random instrument error. This may be determined by evaluation of color and viscosity of the sample extract, evaluation of the chromatogram and reanalysis of the extract

(2) Investigate the spiking volume and concentration of the internal standard if the recovery of surrogate is outside the QC limits. If the wrong spiking volume or wrong concentration of the internal standard can be documented, the internal standard amount may be adjusted to actual spiking amount, and the results recalculated.

If the recovery of the surrogate standard is within the recovery range, then the internal standard method is effectively quantifying the compounds. If the associated surrogate is outside of the recovery range, the internal standard area is affecting quantitation. Evaluate whether this indicates a potential high or low bias for the associated compound results (low IS=high surrogate=high bias; high IS=low surrogate=low bias). Internal standard areas outside of the acceptance limits are qualified with the "*" flag on the recovery summary report form (Form 8), and discussed in the project narrative.

8.3.6 The following are typical analytical sequences:

Initial Batch:

1. PCB Window Mix
2. Ical standard #1
3. Ical Standard #2
4. Ical Standard #3
5. Ical Standard #4
6. Ical Standard #5
7. Initial Calibration Verification (ICV)
8. Method blank
9. LCS
10. Sample #1
11. .....
12. Last sample in this sequence (within 12 h tune period)
13. Closing CCV

Middle/Final Batch

1. PCB Window Mix
2. Beginning CCV
3. Method blank
4. Sample #1
5. .....
6. Last sample in this sequence (within 12 h tune period)
7. Closing CCV

8.4 IDENTIFICATION PROCEDURES

8.4.1 Using the ions shown on **Tables 6-7** for PCBs examine ion current profiles (ICPs) to locate internal standards, surrogate compounds, and PCBs for each isomer group. Use the RRT window data as guidelines for location of PCB isomers. (A reverse search software routine can be used to locate compounds of concern.)

8.4.2 SIM Data -- Obtain appropriate selected ion current profiles (ICPs) for IS quantitation and confirmation ions for the quantitation and confirmation ions for each PCB isomer group.

8.4.3 PCB Analytes

8.4.3.1 For all PCB candidates, confirm the presence of an (M-70) – ion cluster by examining ICPs or spectra for at least one of the most intense ions in the appropriate ion cluster.

8.4.3.2 For Cl_3-Cl_7 isomer groups, examine ICPs or spectra for intense $(M+70)^+$ ions that would indicate a coeluting PCB containing two additional chlorines. (GC retention time data show that this is not a potential problem for other PCB isomer groups.) If this interference occurs, a correction can be made. Obtain and record the area for the appropriate ion (**Table 9**) for the candidate PCB isomer group. Use the information in Table 10 to correct the measured abundance of M+. For example, if a Cl_7-PCB and a Cl_5-PCB candidate coelute, the Cl_7-PCB will contribute to the ion measured for m/z 326 and m/z 324, the quantitation and confirmation ions, respectively, for a Cl_5-PCB. Obtain and record the area for m/z 322 (the lowest mass ion in the $(M+-70)^+$ ion cluster of a Cl_5-PCB fragment produced by a Cl_7-PCB). To determine the m/z 326 and m/z 324 areas produced by the Cl_5-PCB, calculate the Cl_7-PCB contribution to each and subtract it from the measured area. In this example, 164% of the area measured for m/z 322 should be subtracted from the area measured for m/z 324, and 108% of the m/z 322 area should be subtracted from the area measured for m/z 326 (**Table 10**).

8.4.3.3 For Cl_2-Cl_8-PCB candidates, examine ICPs or spectra for intense $(M+35)^+$ ions that would indicate a coeluting PCB containing one additional chlorine. This coelution causes interferences because of the natural abundance of ^{13}C. (This interference will be small and can be neglected except when measuring the area of a small amount of a PCB coeluting with a large amount of another PCB containing one more chlorine.) To correct for this interference, obtain and record the area for the appropriate ion (**Table 11**) from the $(M-1)^+$ ion cluster, and subtract 13.5% of the area measured for the $(M-1)^+$ ion from the measured area of the quantitation ion. For example, for Cl_5–PCB candidates, obtain and record the area for m/z 325; subtract 13.5% of that area from the measured area of m/z 326.

8.4.3.4 All Analytes -- Use ICP data to calculate the ratio of the measured peak areas of the quantitation ion and confirmation ion(s), and compare to the acceptable ratio (**Table 9**). If acceptable ratios are not obtained, a coeluting or partially coeluting compound may be interfering. Examination of data from several scans may provide information that will allow application of additional background corrections to improve the ion ratio.

8.4.4 IDENTIFICATION CRITERIA

8.4.4.1 Internal Standards

8.4.4.1.1 Tetrachlorobiphenyl, 2,2',5,5'-C13 -- the abundance of m/z 303 relative to m/z 305 must maximize simultaneously. The area measured for m/z 303 must be within 30% of the area measured during the most recent continuing calibration.

8.4.4.1.2 Retention time must be within ±10 s of that observed during the most recent acceptable calibration.

8.4.4.2 SIM Data for PCBs

8.4.4.2.1 Absolute retention times of surrogate compounds must be within ±10s of that measured during the last previous continuing calibration check.

8.4.4.2.2 Quantitation and confirmation ions for each PCB isomer group must maximize within ±1 scan of each other.

8.4.4.2.3 The integrated ion current for each quantitation and confirmation ion must be at least three times background noise and must not have saturated the detector.

8.4.4.2.4 For each PCB isomer group candidate, the ratio of the quantitation ion area to the confirmation ion area must be within limits shown on **Table 9**; at least one ion in the (M-70) + ion cluster most be present.

9. Data Reduction and Calculations

9.1 Response Factor Calculation

Calculate five response factors (RFs) for each PCB calibration congener and surrogate compound relative to the IS. RF is a unitless number; units used to express quantities must be equivalent.

$$RF = \frac{A_x \times Q_{is}}{A_{is} \times Q_x} \qquad (1)$$

where:

A_x = integrated ion abundance of quantitation ion for a PCB calibration congener or a surrogate compound.

A_{is} = integrated ion abundance of m/z 303, the quantitation ion for the internal standard.

Q_{is} = injected quantity of the internal standard.

Q_x = injected quantity of PCB calibration congener or surrogate compound.

9.2 The relative standard deviation (%RSD) is calculated using the following equation:

$$\%RSD = \frac{Standard\ Deviation}{Mean\ RF} \qquad (2)$$

where:

$$Standard\ Deviation = \sqrt{\frac{\sum (RF_i - RF_m)^2}{n-1}} \qquad (3)$$

where:

RF_i = each of the response factors
RF_m = mean of the response factors
n = the total number of initial calibration levels

9.3 Response Factor Reproducibility -- For each PCB calibration congener and surrogate compound, calculate the mean RF from analyses of each of the five CALs. The initial calibration is deemed acceptable if the RSD for each PCB calibration congener is less than or equal to 20%. If the 20% RSD criteria are not met, the calibration may be acceptable if either of the following two conditions are met:

(1) The mean RSD of the nine calibration congeners is less than or equal to 20% and the RSD for any of the nine calibration congeners does not exceed 30%.

(2) Linear calibration using a least squares regression: in this case, the analyst may employ a regression equation for the analyte(s) that do not pass the previous evaluation. The regression will produce the slope and intercept from a linear equation as follows:

$$y = mx + b \qquad (4)$$

where:

y = instrument response (peak area)
m = slope
x = concentration of each calibration standard
b = intercept

It is important that the origin (0,0) is not included as the sixth calibration point and that the above equation does not force through the origin.

The linear regression is deemed acceptable if the correlation coefficient $r \geq 0.995$.

If the initial calibration fails to meet any of the criteria stated above, investigate source of problem, reinject the appropriate CAL standard if data indicate a potential bad injection, evaluate instrument response and chromatographic performance and make adjustments

where needed to improve GC/MS performance prior to re-performing the initial calibration. Instrument maintenance is to be recorded in the Instrument Maintenance Logbook. Response factors for the initial calibration will be reported on the initial calibration summary (Form 6). If linear regression is used, this will be identified on the Form 6.

9.4 SIM Analyte Retention Time Reproducibility

PCB determinations - Absolute retention times of PCB congeners #77, #104, #202, and #189 should not vary by more than ±10 s from one analysis to the next. (Retention time reproducibility is not as critical for congeners #1 and #209 as for the other four congeners, which are used to determine when ion sets are changed.)

9.5 Record a spectrum of each CAL component.

10. Quality Assurance/Quality Control and Corrective Actions

10.1 METHOD BLANK – method blanks will be performed at a frequency of one method blank for each extraction batch of up to a maximum of 20 samples.

10.1.1 A method blank must contain the same amount of surrogate standards and internal standards that are added to each sample.

10.1.2 Analyze a method blank before any samples are analyzed.

10.1.3 An acceptable method blank contains no method analyte at a concentration greater than its lower calibration standard concentration (L1)for that PCB homolog and contains no additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte at its RL. If the method blank that was extracted along with a batch of samples is contaminated, data for the entire batch of samples must be evaluated, and reextractions performed where necessary. If the contaminant(s) present in the method blank are not present above the reporting limit in a particular sample, that sample is not affected by the contamination, and data for that analysis may be used. In this circumstance, the sample data will be reported with its associated method blank.

10.1.4 Corrective action for unacceptable method blank – Reanalyze the blank to insure the contamination is not due to carry over from previous standards or samples. Check solvents, reagents, apparatus and glassware to locate and eliminate the source of contamination before any samples are extracted and analyzed. Any compound present in method blank that is also present in samples will be qualified with a "B" flag on sample data sheets reported to the client, and discussed in the project narrative.

10.1.4 When other criteria in Sect. 9 are not met, results for affected analytes must be labeled as suspect to alert the data user of the observed problem. Included among those criteria are: response factor check for each PCB calibration congener and retention time reproducibility for SIM data acquisition.

10.2 INITIAL CALIBRATION VERIFICATION – a standard at the same approximate concentration as the medium-level calibration solution is used as the initial calibration verification (ICV) to demonstrate the accuracy of the primary calibration standards . The initial calibration is deemed acceptable if the analytes' concentrations in the ICV are determined to be within 80%-120% of the true value. The ICV is ordered from Chem Service, or other suitable supplier, different from the supplier of the primary calibration standards. If the ICV is not acceptable, investigate the source of the problem and correct prior to sample analysis.

10.3 SURROGATE SPIKE

10.3.1 Measure the concentration of both surrogate compounds in every sample and blank.

10.3.2 The warning limits for surrogate compounds are 60-140% recovery for sediment extracts, and the action limits are 30-150%. Aqueous samples will use the 60-140% recovery limits as well. Mitkem is spiking two surrogate standards (Tetrachlorobiphenyl, 3,3',4,4'-C13 and Octachlorobiphenyl, 2,2',3,3',4,4',5,5'-C13). Control limits for this program are not based on statistical evaluation of data, but are "generic" limits. The generic limits may be replaced with statistical limits once sufficient program data are available for evaluation. Statistical control limits are generated from at least 30 data points per the procedures in the current revision of Mitkem SOP 80.0010.

10.3.3 If the surrogate recoveries for an analysis are outside of the warning, the data for that analysis will be evaluated and reanalysis and/or reextraction and reanalysis performed where necessary. The evaluation will include whether one or both surrogates are outside of the warning or the action limits, if coeluting peaks interfere with surrogate or internal standard chromatography and integration. If recoveries are above the normal range, and no target compounds are detected in the analysis, the resulting high bias does not negatively impact the data. Manual integration will be performed where necessary, and properly documented according to the current revision of Mitkem SOP 110.008. Based on this evaluation, data for samples with surrogate recoveries outside of the QC limits may be reported, and the recoveries discussed in the report narrative.

10.3.4 Any surrogate recovery outside of the acceptance limits will be flagged on the surrogate summary sheet (Form 2) with an "*". If the sample is reanalyzed and the surrogate standards are within the acceptance criteria for the reanalysis, and the reanalysis is within holding time, only the results of the reanalysis will be reported. If the same surrogate is outside of the QC limits in the reanalysis, both sets of analysis results will be reported to demonstrate matrix-related problems. This will be discussed in the report narrative.

10.4 LABORATORY CONTROL SAMPLE -- A QC reference check standard (LCS) is also prepared and analyzed for Aroclor-1242 at a concentration of 5000 ng/mL in the extract. For sediment/solid samples, sodium sulfate is used for the QC reference check standard (LCS). Calculate the percent

recovery for the Total PCB Aroclor spike and compare to the project limits of 60-140%. As discussed under Surrogate Standards, the control limit is "generic" and is not based on actual analysis data. As sufficient analysis data become available, statistical LCS control limits may be developed and implemented, following discussion with the client. If the percent recovery for the QC reference check standard (LCS) is out of criteria, the analysis is out of the control for that analyte and the problem should be immediately corrected. The entire batch of samples will need to be reanalyzed (Exception: If the LCS recovery is high and there were no associated positive results, then flag the high recovery with an "*" on the Form 3 and address the issue in the report narrative, investigate the issue further prior to the next LCS).

10.5 MS/MSD SAMPLES -- A matrix spike for Aroclor-1242 is to be analyzed at a rate of 1 matrix spike per every 20 samples at a concentration of 5000 ng/mL in the extract.

10.5.1 Analyze one unspiked and two spiked samples. Calculate the percent recovery based on Aroclor concentration of both samples as follows:

A = concentration of spiked sample
B = concentration of unspiked sample (background)
T = known true value of the spike

$$Percent\,\mathrm{Recovery}(p) = \frac{A-B}{T} \times 100\% \qquad (6)$$

Compare the percent recovery calculated with the project limits of 60-140%. If the total PCB concentrations of the matrix spikes are *greater* than four times the calculated sample amount, then the quality control limits should be applied. If the total PCB concentrations of the matrix spikes are *less* than four times the sample, then there are no established limits applicable. If the percent recovery falls outside of the acceptance range for the total PCB used as the spiking analyte, then the matrix spike recovery failed the acceptance criteria. Check for documentable errors *(e.g.,* calculations and spike preparations) and then check the unspiked sample results and surrogate recoveries for indications of matrix effects. If no errors are found and the associated QC reference check standard (Laboratory Control Sample [LCS]) is within 60-140%, then sample matrix effects are the most likely cause. Note this in the report narrative.

A relative percent difference (RPD) must also be calculated on the matrix spike set recoveries. This is calculated as follows:

A = % recovery of matrix spike sample
B = % recovery of matrix spike duplicate sample

$$RPD = [\frac{(A-B)}{(A+B)/2}] \times 100\% \qquad (7)$$

where (A-B) *is taken as an absolute value*

If the total PCB concentrations of the matrix spike set are *greater* than four times the calculated sample amount, then an RPD of 40% or less is acceptable. If the total PCB concentrations of the matrix spike set are *less* than four times the calculated sample amount than there are no established limits applicable to the RPD. If the criterion is not met, check for documentable errors *(e.g.,* calculations and spike preparations) and then check the unspiked sample results and surrogate recoveries for indications of matrix effects. If no errors are found and the associated LCS is within 60-140%, then sample matrix effects are the most likely cause. Note this in the report narrative.

10.6 CALIBRATION - Included among initial and continuing calibration procedures are numerous quality control checks to ensure that valid data are acquired.Continuing calibration checks are accomplished with results from analysis of the medium-level calibration solution and the PCB Window Defining Mixture to monitor criteria times for the five (5) ion sets. Continuing calibration acceptance criteria must be met prior to sample analysis.

10.6.1 If some criteria are not met for a Continuing Calibration Check after a 12-h period during which samples were analyzed (ending CCV), those samples must be reanalyzed. Those criteria are: GC performance, MS calibration as indicated by DFTPP spectrum, and MS sensitivity as indicated by area of internal standards. If the criteria are still not met for the ending CCV for the reanalyses, the condition is due to sample matrix effects. In this circumstance, both sets of data are reported to the client, with the evaluation of the calibration discussed in the report narrative.

10.7 A Method Detection Limit (MDL) study will be performed on a representative instrument in accordance with the procedures described in 40 CFR Part136, Appendix B prior to analysis of samples. Clean sodium sulfate will be used as the matrix for this MDL study for soils/sediments. Detection limits vary among method analytes and with sample matrix, sample preparation procedures, condition of the GC/MS system, type of data acquisition, and individual samples. Detection limits for individual PCB congeners increase with increasing number of chlorine atoms, with the detection limit for decachlorobiphenyl being somewhat higher than that of a monochlorobiphenyl. The detection limit for total PCBs will depend on the number of individual PCB congeners present. SIM data acquisition procedures used for this method reduce the detection limit for PCBs by at least a factor of three.

11. Data Validation and Reporting

11.1 From appropriate ICPs of quantitation ions, obtain and record the spectrum number of the chromatographic peak apex and the area of the entire chromatographic peak.

11.2 GC/MS Analytes Detected Above the Initial Low Level Calibration Standard Concentration

Any individual PCB analyte amount in a sample or QC sample above the initial low level calibration standard concentration (L1) will be reported as such with no associated flags. Any individual PCB analyte amount that exceeds the high level calibration standard (L5) of the calibration curve will require dilution and re-analysis of the extract to place that analyte within the calibration range. The result for any analyte that is reported above the high level calibration standard will be qualified with an "E" flag on the Form 1 data sheet.

11.3 GC/MS Analytes Detected Below the Initial Low Level Calibration Standard Concentration

As stated in Section 1.2, a Method Detection Limit (MDL) will be performed in accordance with procedures set forth in 40CFR Part 136, Appendix B. Any individual PCB analyte amount in a sample or QC sample that is above the established MDL but below the initial low level calibration standard concentration may be reported and appropriately flagged with a "J" flag. A "J" flag signifies that the analyte amount was below the initial low level calibration standard concentration but above the determined MDL for the analyte. Individual PCB analytes will only be "J" flag reported if sufficient ion confirmation information is present. Analytes determined to be not present will be reported as not detected. The L1 Standard Concentration concentration will be reported for that analyte, and qualified with the "U" flag on the Form 1 data sheet.

11.4 PCB Homolog Group Amounts

For each PCB Homolog Group all reportable PCB analytes associated with a given chlorination level (i.e. All dichlorobiphenyls) will be summed and a total provided. No flagging of Homolog Group concentrations will occur. This will provide 10 PCB sub-totals from monochlorobiphenyl to decachlorobiphenyl. If for a given Homolog there are no reportable analytes to report or sum, then a not detected ("U" flagged value) will be reported. The associated L1 Standard concentration value for that chlorination level will be reported as the Homolog reporting limit.

11.5 Total PCB Amount

The Total PCB amount for a sample or QC sample will be provided by summation of the Homolog Group amounts. No flagging of the Total PCB amount will occur. If all 10 Homolog Groups are reported as not detected ("U" flagged values), then the Total PCB amount will be reported as not detected ("U" flagged). For this reporting condition (i.e. Total PCB amount = "U"), the single lowest Initial Low Level Calibration Standard Concentration of the 10 Homolog groups will be used and will provide the reporting limit for the Total PCB amount.

11.6 All soil/sediment results will be reported on a dry-weight basis in ug/Kg. Percent moisture will be reported on the Form 1 data sheet. Tissue samples are reported on a wet-weight basis.

11.7 For PCBs, sum the areas for all isomers identified at each level of chlorination (*e.g.*, sum all quantitation ion areas for Cl_4-PCBs).

11.8 Calculate the concentration of each surrogate compound and PCB isomer group using the formula:

$$C_x = \frac{A_x \times Q_{is} \times V \times DL}{A_{is} \times RF \times W \times D} \qquad (8)$$

where: C_x = concentration (micrograms per kilogram or micrograms per liter) of surrogate compound or a PCB isomer group,

A_x = the area of the quantitation ion for each surrogate compound or the sum of quantitation ion areas for all PCB isomers at a particular level of chlorination,

A_{is} = the area of the internal standard quantitation ion,

Q_{is} = quantity (micrograms) of internal standard added to the extract before GC/MS analysis,

RF= calculated response factor for the surrogate compound or the PCB calibration compound for the isomer group (level of chlorination), and
W= weight (kilograms) of sample extracted.

V = final volume of the extract (ml).

W = weight of sample extracted (Kg)

DL = dilution factor.

D= (100 - % moisture)/100

Use the average RF calculated during Initial Calibration.

11.9 Report calculated values to two significant figures.

11.10 When samples of known composition or fortified samples are analyzed, calculate the percent method bias using the equation:

$$B = \frac{C_s - C_t}{C_t} \times 100\% \qquad (9)$$

where: C_s = measured concentration (in micrograms per kilogram or micrograms per liter) and

C_t = theoretical concentration (*i.e.*, the quantity added to the sample aliquot/weight or volume of sample aliquot).

Note: The bias value retains a positive or negative sign.

11.11 Automated identification and measurement software for PCBs will be used to assist in handling and reducing the data. Mitkem uses instrument manufacturer (Agilent) Enviroquant software to control instrument operation and data acquisition. Mitkem uses the Target (Thermo LabSystems) chromatographic software system for data evaluation and reporting as well as the Omega LIMS system for data reporting.

11.12 Samples may be frozen upon receipt as described in Section 4 and accumulated until sufficient samples are available to create a sample delivery group (SDG). SDGs are typically 20 samples, but this number may be adjusted based on sample collection schedules, instrument operation schedules or data report schedule requirements for the client.

11.13 Sample analysis data will be reported in a full CLP-format data package, as described in the Contract Laboratory Program (CLP) Statement of Work for Organics Analysis, OLM04.2. Forms may be adapted for the SIM method.

12. Health and Safety

12.1 Safety glasses and disposable gloves must be worn when handling samples and extracts.

12.2 All manipulations of sample extracts should be conducted inside a chemical fume hood. The analyst should minimize manipulation of sample extracts outside of a fume hood.

12.3 Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets available in the binder outside of the organic preparation laboratory to familiarize themselves with the precautions of handling applicable solvents and chemicals used to process samples. The analyst should refer to the laboratory Chemical Hygiene Plan for further safety Information.

12.4 Samples remaining after analysis should be disposed of through Mitkem's internal disposal plan. Refer to the standard operating procedures for disposal of laboratory waste.

13. Pollution Prevention; Waste Management; Definitions and Acronyms

See Sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

14. References

Method 680. Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry" November, 1985, Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, USEPA, Cincinnati, OH

Standard Operating Procedure for the Determination of PCBs in Sediment by Gas Chromatography/Mass Spectrometry by EPA Method 680. General Electric (GE) Hudson River Design Support, Sediment Sampling and Analysis Program, Revision 1, July 16, 2002.

Gebhart, J. E., Hayes, T. L., Alford-Stevens, A. L., and W. L. Budde, "Mass Spectrometric Determination of Polychlorinated Biphenyls as Isomer Groups", Anal. Chem. 57, 2458, 1985.

Ballschmiter, K. and M. Zell, Fresenius Z. Anal. Chem., 302, 20, 1980.

Table 1. Specific GC/MS Operating Parameters

Injector:

GC PAL, CTC Analytics
Injection mode: Pulse Splitless
Syringe Size: 10 uL
Injection Volume: 4 uL
Solvent Flush: Yes, Solvent A and B
Solvent for rinsing syringe: Hexane
Syringe Wash: 2 pumps each
Plunger Speed: Fast
Viscosity Delay: 1 second
Sample Pumps: 4

Heating Block: 300 degrees C.
Carrier Gas: He
Pressure: 24psi until 1.8 min; then constant flow at 0.6 ml/min
Total Flow: 50mL/min.

Column:

DB-5 (Agilent): 60M x 0.25mm x 0.25um

Oven:

HP 7890 Series GC System
Model: G2614A

Ramp	Rate		Next Hold time	Run Time
1		80	2.0min	2.0min
2	75deg/min	160	0.0min	3.07min
3	8 deg/min	285	16.0min	34.7min

Note: GC oven temperature program or carrier gas flow rate may be adjusted if necessary to improve chromatographic resolution

Detector:

Agilent 5973 or 5975 Mass Selective Detector (MSD)

MSD Transfer Line Heater: 300 °C
Temperature for Quadrapole: 150 °C
Ion Source: 250 °C
Emission Current: 20 microamps
Scan Time: 0.60 seconds
Ionization Mode: Electron Ionization

Table 2. PCB Congeners Used as Calibration Standards

PCB Isomer Group	Congener Number[a]	Chlorine Substitution
Concentration Calibration Standard		
Monochlorobiphenyl	1	2
Dichlorobiphenyl	5	2,3
Trichlorobiphenyl	29	2,4,5
Tetrachlorobiphenyl	50	2,2',4,6
Pentachlorobiphenyl	87	2,2',3,4,5'
Hexachlorobiphenyl	154	2,2',4,4',5,6'
Heptachlorobiphenyl	188	2,2',3,4',5,6,6'
Octachlorobiphenyl	200	2,2',3,3',4,5',6,6'
Nonachlorobiphenyl[b]	---	---
Decachlorobiphenyl	209	2,2',3,3',4,4',5,5',6,6'
Retention Time Calibration Standards		
Tetrachlorobiphenyl	77	3,3',4,4'
Pentachlorobiphenyl	104	2,2',4,6,6'
Heptachlorobiphenyl	189	2,3,3',4,4',5,5',
Octachlorobiphenyl	202	2,2',3,3',5,5',6.6'

[a] Numbered according to the system of Ballschmiter and Zell (2).
[b] Decachlorobiphenyl is used as the calibration congener for both nona- and decachlorobiphenyl isomer groups.

Table 3. Composition and Approximate Concentrations of Calibration Solutions For SIM Data Acquisition for PCB Determinations

	Concentration (ng/mL)				
Compound	**CAL 1**	**CAL2**	**CAL 3**	**CAL 4**	**CAL5**
Cal. Congeners					
Cl_1 (#1)	10	50	250	1000	2500
Cl_2 (#5)	10	50	250	1000	2500
Cl_3 (#29)	10	50	250	1000	2500
Cl_4 (#50)	20	100	500	2000	5000
Cl_5 (#87)	20	100	500	2000	5000
Cl_6 (#154)	20	100	500	2000	5000
Cl_7 (#188)	30	150	750	3000	7500
Cl_8 (#200)	30	150	750	3000	7500
Cl_{10} (#209)	50	250	1250	5000	12500
Internal Standard					
Cl_4-C13-IS	800	800	800	800	800
Surrogate Compounds					
Cl_4-C13	10	50	250	1000	2500
Cl_8-C13	10	50	250	1000	2500

Table 4. Retention Time Calibration Standards
Concentration (ng/µL)

Compound	CAL 1
Pentachlorobiphenyl (#014)	2.50
Tetrachlorobiphenyl (#77)	2.50
Octachlorobiphenyl (#202)	2.50
Heptachlorobiphenyl (#189)	2.50

Table 6.
Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Sets of ≤ 27 Ions Each

Ion Set	Isomer Group/ IS/Surrogate	Quant.Ion	Confirm Ions	M-70 Ions	M+70 Ions	M+35 Ions	Ion Measured[a] for Correction	
1	Cl_1	188	190	152,153[b]	256,258	222,224	-	-
	Cl_2	222	224	152,153,186,188[c]	290,292,294	256,258	-	221
	Cl_3	256	258	186,188	-	290,292,294	-	225
	Cl_4	292	290,294	220,222	-	-	-	-
2	Cl_3	256	258	186,188	324,326,328	290,292,294	254	255
	Cl_4	292	290,294	220,222	360,362	324,326,328	288	289
	Cl_5	326	324,328	254,256,258	-	360,362	-	323
	Cl_6	360	358,362	288,290,292	-	-	-	-
3	Cl_5	326	324,328	254,256	392, 394,396,398	360,362	322	323
	Cl_6	360	358,362	288,290	-	392,394,396,398	-	357
	Cl_7	394	392,396	322,324,326	-	-	-	-
4	Cl_6	360	358,362	288,290	426,428,430,432	392,394,396	356	357
	Cl_7	394	392,396,398	322,324	-	428,430,432	-	391
	Cl_8	430	428,432	356,358,360	-	462,464,466	-	425
	Cl_9	464	460,462,466	390,392,394	-	-	-	-
5	Cl_8	430	426,428,432	356,358,360	494,496,498,500	462,464,466	-	425
	Cl_9	464	460,462,466	390,392,394	-	496,498,500	-	-
	Cl_{10}	498	494,496,500	424,426,428,430	-	-	-	-
	Chrysene-d_{12}	240	241	-	-	-	-	-

[a]See Tables 9-10.
[b]Cl_1-PCBs lose HCl.
[c]Some Cl_2-PCBs lose Cl_2 and some lose HCl.

Table 7. Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Ion Sets of ≤ 27 Ions

Ion Set #1[a]	Ion Set #2[b]	Ion Set #3[c]	Ion Set #4[d]	Ion Set #5[e]
152	186			356
153	188			358
186	220	254	288	360
187	222	256	290*	390
188	254	288	322	392
189	255	290*	324	394
190	256	322	326	424
220	258	323	356	425
221	288	324	357	426
222	289	326	358	428**
224	290*	328	360	430
255	292	357	362	432
256	294	358	390	462
258	323	360	391	464
290*	324	362	392	466
292	326	392	394	496
294	328	394	396	498
	358	396	398	499
	360	398	425	500
	362		426	502
			428**	
			430	
			432	

			460	
			462	
			464	
			466	

- *** Also scan for 303/305 for Cl_4 Internal Standard and surrogate**
- **** Also scan for 440/442 for Cl_8 surrogate**

Table 7. Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Ion Sets of ≤ 27 Ions (Cont.)

Scan Range #1	Scan Range #2	Scan Range #3	Scan Range #4	Scan Range #5
145m/z to 330m/z	179m/z to 398m/z	240m/z to 428m/z	233m/z to 520m/z	349m/z to 532m/z
SIS Ion Preparation	**SIS Ion Preparation**	**SIS Ion Preparation**	**SIS Ion Preparation**	**SIS Ion Preparation**
Mass Range 1: 150mz/ to 155m/z	Mass Range 1: 184mz/ to 226m/z	Mass Range 1: 244mz/ to 260m/z	Mass Range 1: 236mz/ to 296m/z	Mass Range 1: 350mz/ to 366m/z
Mass Range 2: 180mz/ to 195m/z	Mass Range 2: 250mz/ to 263m/z	Mass Range 2: 284mz/ to 294m/z	Mass Range 2: 316mz/ to 368m/z	Mass Range 2: 384mz/ to 400m/z
Mass Range 3: 215mz/ to 260m/z	Mass Range 3: 283mz/ to 300m/z	Mass Range 3: 318mz/ to 332m/z	Mass Range 3: 385mz/ to 404m/z	Mass Range 3: 418mz/ to 438m/z
Mass Range 4: 285mz/ to 300m/z	Mass Range 4: 320mz/ to 332m/z	Mass Range 4: 388mz/ to 404m/z	Mass Range 4: 422mz/ to 438m/z	Mass Range 4: 456mz/ to 472m/z
Mass Range 5: not used	Mass Range 5: 355m/z to 368m/z	Mass Range 5: not used	Mass Range 5: 455m/z to 506m/z	Mass Range 5: 490m/z to 508m/z
R.T. Window (minutes)	**R.T. Window (minutes)**	**R.T. Window (minutes)**	**R.T. Window (minutes)**	**R.T. Window (minutes)**
5.00 to 17.21	17.21 to 25.01	25.01 to 30.25	30.25 to 36.35	36.35 to 54.67
Total number of Ions	**Total number of Ions**	**Total number of Ions**	**Total number of Ions**	**Total number of Ions**
17	20	19	27	20

[a] Ions to identify and measure Cl_1 – Cl_4 PCBs

[b] Ions to identify and measure Cl_3– Cl_6 PCBs

[c] Ions to identify and measure Cl_5 – Cl_7 PCBs.

[d] Ions to identify and measure Cl_6 – Cl_9 PCBs

[e] Ions to identify and measure Cl_8 – Cl_{10} PCBs

Table 8. Known Relative Abundances of Ions in PCB Molecular Ion Clusters [a]

m/z	Relative Intensity
Monochlorobiphenyls	
188	100.00
189	13.50
190	33.40
192	4.41
Dichlorobiphenyls	
222	100.00
223	13.50
224	66.00
225	8.82
226	11.20
227	1.44
Trichlorobiphenyls	
256	100.00
257	13.50
258	98.60
259	13.20
260	32.70
261	4.31
262	3.73
263	0.47
Tetrachlorobiphenyls	
290	76.20
291	10.30
292	100.00
293	13.40
294	49.40
295	6.57
296	11.00
297	1.43
298	0.95
Pentachlorobiphenyls	
324	61.00
325	8.26
326	100.00
327	13.50
328	65.70
329	8.78
330	21.70
331	2.86
332	3.62
333	0.47
334	0.25

m/z	Relative Intensity
Hexachlorobiphenyls	
358	50.90
359	6.89
360	100.00
361	13.50
362	82.00
363	11.00
364	36.00
365	4.77
366	8.92
367	1.17
368	1.20
369	0.15
Heptachlorobiphenyls	
392	43.70
393	5.91
394	100.00
395	13.50
396	98.30
397	13.20
398	53.80
399	7.16
400	17.70
401	2.34
402	3.52
403	0.46
404	0.40
Octachlorobiphenyl	
426	33.40
427	4.51
428	87.30
429	11.80
430	100.00
431	13.40
432	65.6
433	8.76
434	26.90
435	3.57
436	7.10
437	0.93
438	1.18
439	0.15
440	0.11

m/z	Relative Intensity
Nonachlorobiphenyls	
460	26.00
461	3.51
462	76.40
463	10.30
464	100.00
465	13.40
466	76.40
467	10.20
468	37.60
469	5.00
470	12.40
471	1.63
472	2.72
473	0.35
474	0.39
Decachlorobiphenyl	
494	20.80
495	2.81
496	68.00
497	9.17
498	100.00
499	13.4
500	87.30
501	11.70
502	50.00
502	6.67
504	19.70
505	2.61
506	5.40
507	0.71
508	1.02
509	0.13

[a] Source: Rote and Morris (7)

Table 9. Quantitation, Confirmation, and Interference Check Ions for PCBs, Internal Standards, and Surrogate Compounds

Analyte/ Internal Std.	Nom. MW	Quant. Ion	Confirm. Ion	Expected Ratio [a]	Accept Ratio [a]	M-70 Confirm. Ion	Interference Check Ions M+70	M+35
PCB Isomer Group								
Cl_1	188	188	190	3.0	2.5-3.5	152[b]	256	222
Cl_2	222	222	224	1.5	1.3-1.7	152	292	256
Cl_3	256	256	258	1.0	0.8-1.2	186	326	290
Cl_4	290	292	290	1.3	1.1-1.5	220	360	326
Cl_5	324	326	324	1.6	1.4-1.8	254	394	360
Cl_6	358	360	362	1.2	1.0-1.4	288	430	394
Cl_7	392	394	396	1.0	0.9-1.2	322	464	430
Cl_8	426	430	428	1.1	0.9-1.3	356	498	464
Cl_9	460	464	466	1.3	1.1-1.5	390	-	498
Cl_{10}	494	498	500	1.1	0.9-1.3	424	-	-
Internal standard								
Cl_4-C13		303	305					
Surrogate Compounds								
Cl_4-C13		303	305					
Cl_8-C13		440	442					

[a] Ratio of quantitation ion to confirmation ion.

[b] Monochlorobiphenyls lose HCl to produce an ion at m/z 152.

Table 10. Correction for Interference of PCB Containing Two Additional Chlorines

Candidate Isomer Group	Quant Ion	Confirm. Ion	Ion Measured to Determine Interference	% of Meas. Ion Area to be Subracted from Quant. Ion Area	Confirm. Ion Area
Trichlorobiphenyls	256	258	254	99%	33%
Tetrachlorobiphenyls	292	290	288	65%	131%
Pentachlorobiphenyls	326	324	322	108%	164%
Hexachlorobiphenyls	360	362	356	161%	71%
Heptachlorobiphenyls	394	396	390	225%	123%

Table 11. Correction For Interference of PCB Containing One Additional Chlorine

Candidate Isomer Group	Quant Ion	Ion Measured to Determine Interference	% of Meas. Ion Area to be Subracted from Quant. Ion Area
Dichlorobiphenyls	222	221	13.5%
Trichorobiphenyls	256	255	13.5%
Tetrachlorobiphenyls	292	289	17.4%
Pentachlorobiphenyls	326	323	22.0%
Hexachlorobiphenyls	360	357	26.5%
Heptachlorobiphenyls	394	391	30.9%
Octachlorobiphenyls	430	425	40.0%

Figure 1. Merged Ion Current Profile of PCB Calibration Congeners

Quantitation Report

Data File : O:\ORGANIC\SVOA\S5.I\080424.B\S5A1281.D Vial: 2
Acq On : 24 Apr 2008 10:46 am Operator: TM
Sample : SSTD0L35B,SSTD0L35B Inst : S5
Misc : 2,,SSTD025,3| 680 CCV L3□ Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Apr 24 12:13 2008 Quant Results File: S5680_EQ.RES

Method : O:\ORGANIC\SVOA\S5.I\080425.B\S5680_EQ.M (RTE Integrator)
Title : Method 680
Last Update : Thu Apr 24 12:14:48 2008
Response via : Initial Calibration

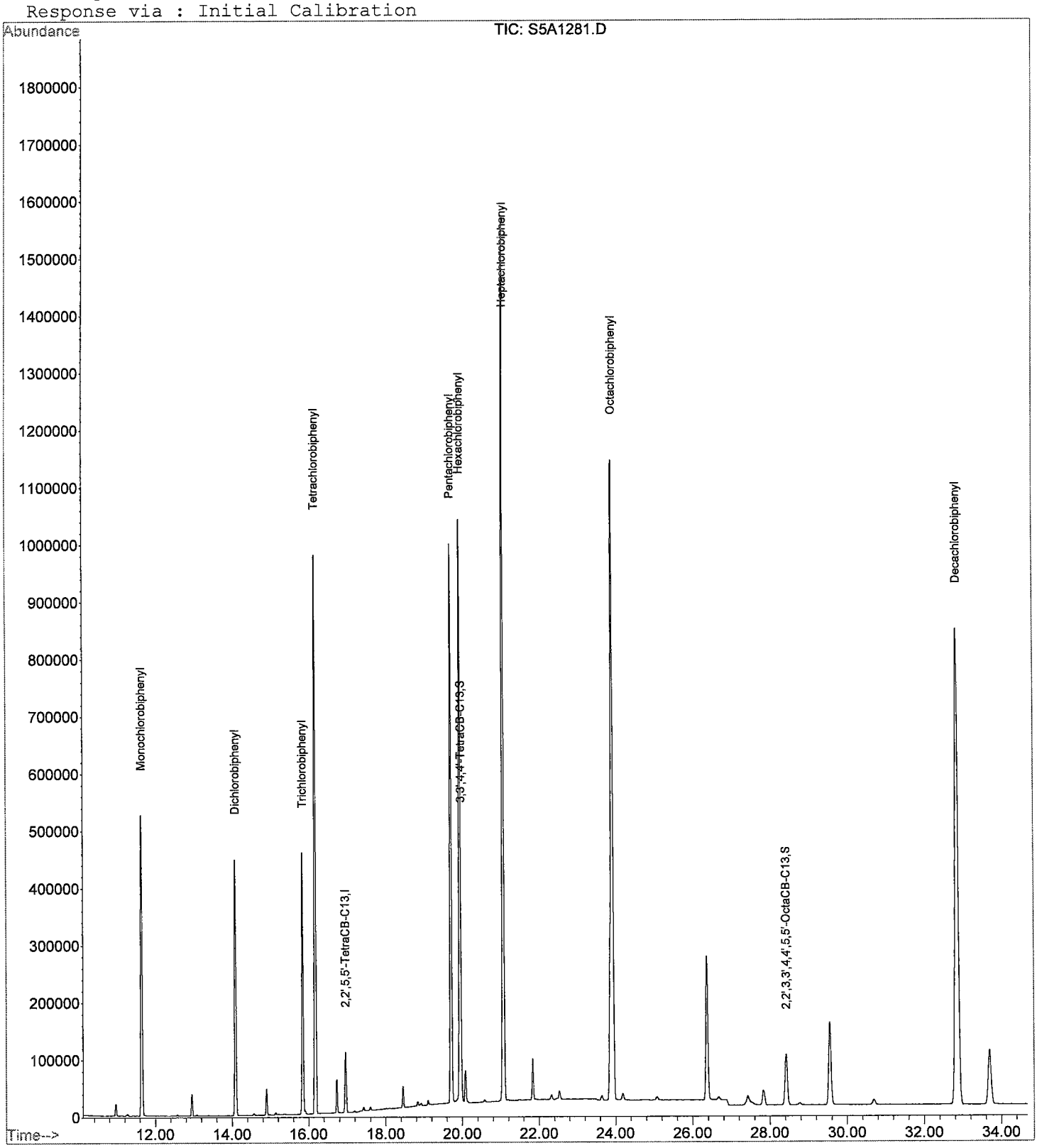
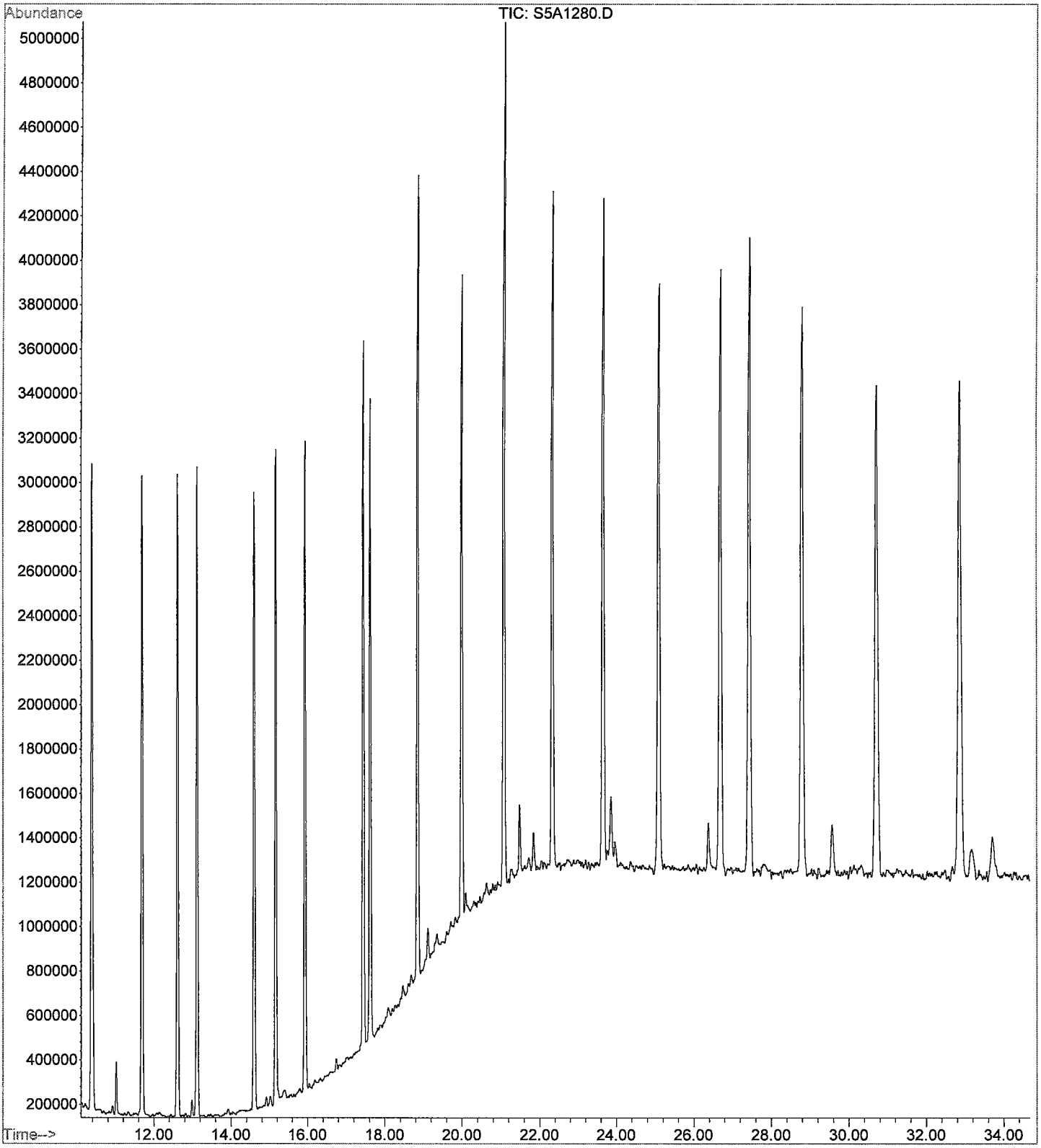


Figure 2. Total Ion Chromatogram of PCB Window Defining Mixture Standard

File : O:\ORGANIC\SVOA\S5.I\080424.B\S5A1280.D
Operator : TM
Acquired : 24 Apr 2008 10:08 am using AcqMethod S5_680FS
Instrument : S5
Sample Name: SSTDWIN5B,SSTDWIN5B
Misc Info : 3,,WINMIX,3
Vial Number: 1



MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Determination of Metals in Water and Soil by Inductively Coupled Argon Plasma Atomic Emission Spectrometry using Method SW846 6010C

Rev. 12

	Signature	Date
QA Director:	Sharon B Lauh	2/3/09
Lab Director:	Kin Chui	2/3/09

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Determination of Metals in Water and Soil
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1. Scope and Application

This SOP describes the procedures applicable to the analysis of the elements listed in **Attachment 1**. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. **Section 8.1** provides the Method references for sample digestion procedures. See **LIMS Test Information/ Test/Limits** for analytes and their associated MDL/PQLs.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 Prior to analysis, samples must be digested using appropriate sample preparation methods.

3.2 The method measures element specific emitted light by optical spectrometry. The samples are nebulized and the resulting aerosol is transported to the plasma torch. The metals pass through the hot zone of the plasma, where they take up energy. Subsequently the metals pass through the cold zone (relatively) of the plasma where they give up the excess energy at element specific wavelength. The spectra are dispersed by a grating spectrometer, and the intensity of the emitted light is measured by a solid state photomultiplier. Background correction is required for trace element determination. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the

analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured.

4. Sample Preservation, Containers, Handling, and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For metals analysis by Method 6010C, water samples are collected in 500 ml plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Soils are collected in 8-ounce glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 Soil samples are stored at 4°C ± 2°C until analyzed.

4.3 Sample holding time for metals analysis by method 6010C is 180 days from the date of sample collection for both water and soil.

5. Interferences and Potential Problems

Several types of interference effects may contribute to inaccuracies in the determination of an analyte by ICAP-AES.

5.1 Spectral interferences - Can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena. The first of these can be compensated for by utilizing a computer correction of raw data, requiring monitoring and measurement of the interfering element. The second effect may require selection of an alternative wavelength. In addition one could select an alternate wavelength where interference is minimal or absent. The 4300DV and the 3100XL used at Mitkem have many spectral lines from which to choose. The third effect can usually be compensated by a background correction adjacent to the analyte line.

5.2 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 salts or high acid concentrations. If physical interferences are present, they must be reduced by such means as a high-salts nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved salts is a salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem can be controlled by a high-salts nebulizer, wetting the argon prior to nebulization, using a tip washer, or by diluting the samples. A mass flow controller is used to control the argon gas flow rate.

5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. These effects can be minimized by careful selection of

operating conditions, by buffering of the sample, by matrix matching, and by standard-addition procedures.

5.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer or from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with the rinse solution between samples. This method requires a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed using a longer rinse period.

5.5 Physical, chemical and spectral interferences are primarily attributed to the sample matrix. If interference caused by a particular sample matrix is known, in many cases it can be circumvented. However, when the nature of the sample is unknown, following tests can be used to ensure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.

5.5.1 Dilution Test -If the analyte concentration is sufficiently high (minimally a factor of 10x above the method detection limit (MDL)), an analysis of a 1:5 dilution should agree within ±10% of the original determination. If not, a chemical or physical interference effect could be suspected.

5.5.2 Post Digestion Spike Addition - If the matrix spike (pre-digestion) recovery falls outside of the control limits (75% - 125%), matrix interference is suggested. In this case an analyte spike is added to a portion of a prepared sample or its dilution at a level just below the mid-point of the calibration curve. Post digestion spikes should be recovered to within 80% - 120% of the known value. If not, a matrix effect should be suspected.

5.5.3 Comparison with alternative method analysis-when investigating a sample matrix, comparison tests may be performed with other analytical techniques, such as atomic absorption spectrometry, or ICP-mass spectrometry. This should only be done after consultation with the client.

5.5.4 Internal Standard Addition technique can be used (Refer to Section 4.4.2 of Method 6010C.)

6. Equipment and Apparatus

6.1 Inductively coupled argon plasma emission spectrophotometer (ICAP).

The ICAPs used at Mitkem are a Perkin-Elmer Model 4300DV and a 3100XL. The 4300DV is outfitted with an AS-93plus, 157-position autosampler and a high precision, three channel, peristaltic pump. The 3100XL is outfitted with an AS-91, 160-position

autosampler and a high precision, three channel peristaltic pump. Both ICPs have axial viewing capability, as compared to the more traditional radial viewing ICAP. Axial viewing provides greater sensitivity for all elements analyzed. The solid state detector is capable of analyzing at approximately 6000 wavelengths.

The built-in radio-frequency generator is FCC compliant.

The systems are computer controlled through a 32-Bit, Microsoft Windows NT operating system. This system allows for great flexibility in controlling the instrument.

The required high purity argon gas is piped in from a main storage tank that is located in the rear of the building. The gas is stored in liquid form, drawn-off as a gas and then piped into the building for distribution to the instruments. The liquid argon supply is monitored remotely by the supplier, and resupplied on an automatic-delivery basis.

6.1.1 Operating conditions - The analyst should follow the instructions provided by the instrument manufacturer. Instrument detection limits, linear dynamic ranges, and interference effects are established for each analyte line used. All measurements must be in the instrument linear range where spectral interference correction factors are valid. 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.

6.1.2 Daily emissions of the highest standard for arsenic, copper, lead and selenium are recorded as a means to ensure the plasma is stable before analysis and also to chart potential problems especially with the power tube, sample introduction system, or RF generator. The normal range of the four analytes above are (the unit is intensity generated by instruments):

Optima 2 (3100 XL)	Optima 3 (4300 DV)
As 2,000 - 3,000	As 2,000 - 2,500
Cu 600,000 - 900,000	Cu 400,000 - 600,000
Pb 12,000 - 20,000	Pb 4,000 - 8,000
Se 2,000 - 4,000	Se 1,800 - 2,400

6.1.3 For analysis of normal environmental samples use the following standard operating conditions:

Wattage: 1450 – 1500
Argon flow rate (L/min): 15
Nebulizer flow rate (L/min): 0.55
Sample flow rate (mL/min): 2
Rinse time (sec): 60
Read delay (sec): 60

6.1.4 For regular day-to-day ICAP measurements, the plasma need not be optimized prior to analysis. The plasma needs to be optimized each time a major change occurs in instrument configuration, such as replacement of the torch and/or maintenance, unless response is still within normal ranges. Responses and alignment are routinely checked after torch cleaning or replacement with the analysis of a 1 mg/L Mn solution for axial view calibration and 10 mg/L Mn solution for radial view calibration. "Optimization" is a function of a series of procedures; refer to the Mitkem SOP 100.0006 for additional information on how to perform general maintenance and troubleshooting procedures.

6.1.5 To optimize the plasma, follow the instructions in the Optima Software Guide.

6.2 ICAP Instrument Maintenance:

6.2.1 Preventative Maintenance:

- Peristaltic pump tubing will be replaced every 16 hours of instrument time or sooner if memory effects are manifested.
- The plasma torch is cleaned using concentrated HNO_3 when torch and sample injector buildup is noted.
- The spray chamber and nebulizer are cleaned approximately every month as needed. Replacement is done when needed.
- Air filters are cleaned once every two weeks or as needed upon visual inspection.
- The RF coil, window and cone are cleaned every 2-3 weeks.
- The instrument undergoes extensive maintenance by a manufacturer's service engineer as needed.

6.2.2 Troubleshooting:

6.2.2.1 Sudden drop in CCV concentration occurs in a run:

- Check to see that sample introduction system (tubing) has not become disconnected.
- Check to see if a clog has occurred in the sample introduction system.
- Check CCV Standard to ensure it is not empty.
- Check room temperature to see if a fluctuation has occurred.

- Recalibrate and rerun if necessary.

6.2.2.2 CCVs drift up and down:

- Remake the CCV standard.
- Check room temperature to see if a fluctuation has occurred.
- If problem persists, call Perkin-Elmer. The problem is usually indicative of a power tube failure.

6.2.2.3 Plasma goes out during analysis:

- Sample is not reaching spray chamber (tubing came apart or there is a clog)
- High levels of salt in the sample caused plasma temperature changes.
- The sample injector is coated with high salt material and needs to be changed.
- The power tube is going (after all others fail repeatedly) and Perkin-Elmer must be called for service.

6.3 Glassware:

6.3.1 Class A volumetric flasks: 100 mL and 50 mL.

6.3.2 Class A volumetric pipettes:

6.3.3 10-100µL adjustable Eppendorf.

6.3.4 100-1000 µL adjustable Eppendorf.

6.3.5 5mL adjustable Fisher Scientific.

6.3.6 100µL and 1mL fixed Wheaton.

6.3.7 10 mL Wheaton ICP tubes.

7. Reagents and Standards

All standard solutions (multi-, and single element), and second source QC solutions are purchased from outside vendors (Perkin-Elmer, Accustandard, Inorganic Ventures, ERA, and High Purity Standards, Inc.). All these solutions are traceable and meet with Mitkem's high purity requirements. ***Please note that standards and reagents from other vendors could be***

used as long as the standards are of high purity (>96%0 and traceable to reference materials. All standards are labeled as instructed in Mitkem SOP # 80.0001 Standard Preparation, Equivalency and Traceability.

7.1 Hydrochloric acid (conc.), HCl, Trace Grade, Fisher Scientific.

7.2 Nitric acid (conc.), HNO_3 , Trace Grade, Fisher Scientific.

7.3 Reagent water (ASTM Type II water). Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. Reagent water is also referred to as DI water.

7.4 Mixed calibration standard solutions. Mixed calibration standards, obtained from High Purity Standards, come in three stock solutions. Calibration standards and QC solutions are made up on a daily basis.

7.4.1 CLP CAL 1 consists of two solutions:

- CLP CAL 1B contains silver only.
- CLP CAL 1A contains all other elements other than antimony, arsenic, cadmium, lead, thallium, and selenium.

7.4.2 CLP CAL 2 contains antimony only.

7.4.3 CLP CAL 3 contains arsenic, cadmium, lead, thallium, and selenium.

7.5 High standard (**ICP Standard 1**):

- Pipet 1mL HNO_3 (conc) and 1.5mL HCl (conc) into a 100mL volumetric flask.
- Pipet 1mL each of CLP CAL 1A & 1B stock solution and 0.1mL each of intermediate CLP CAL 2 and 3 into the flask.
- Pipet 0.1mL of Titanium single element standard into the flask.
- Bring to volume with DI water.

7.6 Second (middle) standard (**ICP Standard 2**):

- Pipet 5mL of a 1% HNO_3 and 1.5% HCl acid mixture into a 10 mL ICP tube.
- Add 5mL of the ICP Standard 1.

7.7 Third (low) standard (**ICP standard 3**):

- Pipet 10mL of 1% HNO_3 and 1.5% HCl solution into a 10 mL ICP tube.
- Withdraw 100µL.
- Add 100µL of ICP Standard 1 and mix well.

See **LIMS Test Information/ Test/SPEC** for concentrations of the Initial Calibration Standards.

7.8 **Second source** ICV/CCV standards (CCV100XCONC and Antimony (Sb) Stock solutions) are obtained from Accustandard.

- Pipet 1mL of HNO_3 (conc.) and 1.5mL HCl (conc.) into a 100mL volumetric flask.
- Pipet 1.5mL (for ICV) of CCV100XCONC stock solution into the flask.
- Pipet 0.15mL of Antimony (Sb) Stock solution into the flask.
- Bring to volume with DI water.
- Transfer the solution to a 50mL plastic ICP tube.
- This standard is prepared as needed (in amounts consistent with the ratios above) usually every 1 – 2 days.

See **LIMS Test Information/ Test/SPEC** for analytes, concentrations and recovery limits for the ICV/CCV.

7.9 QC Standards ICSA and ICSB solutions are obtained from High Purity Standards.

ICSAB solution: In addition to the normal element composition of the ICSA solution, sodium, potassium, lead and selenium are added to the ICSAB solution. These stock solutions are also obtained from High Purity Standards.

7.9.1 Pipet 1mL of HNO_3 (conc.) and 1.5mL HCl into a 100mL volumetric flask.

7.9.2 Pipet 1mL of ICSB stock solution (ANALCS-R) into the flask.

7.9.3 Pipet 10mL of ICSA (CLP-INF-1) stock solution into the flask.

7.9.4 Pipet 0.5mL of the sodium/potassium stock solution (INFCS-5) into the flask.

7.9.5 Pipet 1mL of 45mg/L Pb intermediate standard solution into the flask.

7.9.5.1 The intermediate is prepared by adding 4.5mL of Pb at 1000mg/L (#100028-1) up to 100mL with 1%HNO_3 and 1.5% HCL acid solution in a volumetric flask.

7.9.6 Pipet 1mL of 45mg/L Se intermediate solution into the flask.

7.9.6.1 The Selenium intermediate standard is prepared by adding 4.5mL of Se at 1000mg/L (#031) up to 100mL with 1% HNO_3 and 1.5% HCl acid solution in a volumetric flask

7.9.7 Bring to volume with DI water.

7.9.8 Transfer the solution to a 10 mL ICP tube.

See **LIMS Test Information/ Test/SPEC** for concentrations of the ICSA and ICSAB.

7.10 Laboratory control samples (LCS) and Matrix spikes: The LCS standards for soils and waters are obtained from High Purity Standards.

- LCS/Spike standard 1 is prepared by adding 455uL of CLP-CV-1 to the digestion tube.
- LCS/Spike standard 2 and 3 are prepared by adding 45.5uL each of CLP-CV-2 and CLP-CV-3 to the digestion tube.

See **LIMS Test Information/ Test/SPEC** for final concentrations of the LCS/MS.

7.11 Linear Dynamic Range

The stock linear dynamic range (LDR) standards are purchased from Ultra Scientific, Inorganic Ventures and High Purity. The LDR is determined as the highest concentration of standard in which the determined value is within 10% of the true value.

- *LDR Standard I* is prepared by addition of 1 mL of Pb at 1000 mg/L, volumized to 10 ml with 1% HNO3 and 1.5% HCl. Final concentration 100mg/L.
- *LDR Standard II* is prepared by addition of 5 mL of mix QCS-19 and 0.5mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 50 mg/L.

- *LDR Standard III* is prepared by addition of 2.5 mL of mix QCS-19 and 0.25 mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 25 mg/L.

- *LDR Standard IV* is prepared by addition of 1.0 mL of mix QCS-19 and 0.1 mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 10mg/L.

- *LDR Standard V* is prepared by addition of 0.5 mL of mix QCS-19 and 0.05ml of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 5mg/L.

- *LDR Standard VI* is prepared by addition of 0.5 mL of Al and 0.5ml of Mg at 10,000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 500mg/L.

- *LDR Standard VII* is prepared by addition of 3.0 mL of Fe at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 300mg/L.

- *LDR Standard VIII* is prepared by addition of 0.5 mL of Ca at 10,000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 500mg/L.

- *LDR Standard IX* is prepared by addition of 0.5 mL of mix INFCS-5 (High Purity) and volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 250mg/L.

8. Procedure

8.1 The methods in SW-846 for sample digestion or preparation are as follows:

8.1.1 Method 3005, SOP No.100.0003, prepares ground water and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with hydrochloric and nitric acids prior to metal determination.

8.1.2 Method 3010, SOP No.100.0003, prepares aqueous samples, mobility-procedure extracts and waste samples that contain suspended solids for total metal determination by ICP. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid.

8.1.3 Method 3050, SOP No.100.0104, prepares solid waste samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrochloric acid.

8.2 The wavelengths and background correction locations in the reference method can be substituted if they can provide the needed sensitivity and are corrected for spectral interferences. The analyst should follow the instrument manufacturer's instructions, and if possible, approximate the recommended operating conditions.

For each analyte there are a number of possible wavelengths at which analyses could be made. The wavelengths used were selected based on consultations with the ICP specialists at Perkin-Elmer and our own experience.

8.3 Background correction factors are obtained by aspirating a concentrated solution, usually about 200-500ppm, of the interfering element, and measuring the resulting concentrations at the wavelength used. The background correction factors for that interfering element are obtained by dividing the measured concentrations by the actual concentration. For example: by aspirating a 500ppm solution of aluminum, one measures a cadmium concentration 0f 0.010ppm. The aluminum correction factor for cadmium is (0.010)/500 = 0.000020. This means that if the aluminum concentration in a sample is, say, 120ppm, one must subtract 120*0.000020 = 0.0024ppm from the measured cadmium concentration in that sample.

8.4 Interelement Correction (IEC) factors are established quarterly (or biannually at a minimum), for the major salts: aluminum, calcium, iron, magnesium, and also for chromium, copper, manganese, nickel, thallium, titanium and vanadium. Correction factors should not exceed 20% difference from the previous IEC values.

8.5 Linear Dynamic Range is performed quarterly (or biannually at a minimum). Following instrument calibration, solutions with varying concentrations (**section 7.5.13**) of each analyte (LDR standards) are analyzed. The highest concentration, within ±10 % of the true value, establishes the linear range.

8.6 Allow the plasma to become thermally stable before beginning the analyses. This usually requires at least 30 minutes. If the plasma is (for whatever reason) extinguished and needs to be re-lit, subsequent re-stabilization of the plasma takes only 15 minutes provided the plasma is re-lit immediately after it is extinguished.

8.7 The nebulizer flow rate used is constant for all aqueous sample extracts and needs not be reset prior to analysis.

8.8 The instrument operating conditions finally selected as being optimum should provide the lowest reliable instrument detection limits (IDL). (See **Section 10** for IDL calculation)

8.9 The calibration curve consists of a blank (S0) and at least 3 calibration standards. The low concentration calibration standard concentration is less than or equal to the method reporting limit (MRL/LOQ). Sample concentrations less than the lowest standard can only be reported after method modifications have taken place, or with estimated results flagging. The highest concentration must be within the instrument's linear dynamic

range. The standards are run in a sequence from high concentration to low concentration.

8.10 The minimum correlation coefficient for the calibration curve is 0.998. If a correlation coefficient less than 0.998 is obtained, the calibration must be repeated.

8.11 A mid-range ICV (Initial Calibration Verification) Standard from an independent source than the calibration standards) precedes the analysis of the samples. Recovery limits for the ICV are ± 10% of the true value. If the ICV recoveries do not meet acceptance criteria, corrective action must be taken and/or the calibration must be repeated. An ICV can be reanalyzed only once prior to corrective action.

8.11.1 A low level ICV (not required to be second source) is run to verify the lower level of calibration. Recovery limits for the low level ICV are + 30% of the true value. The low level ICV is the same concentration as the lowest standard in the initial calibration (S3), which is at the reporting limit. If the low level ICV recoveries do not meet acceptance criteria, corrective action must be taken and/or the calibration must be repeated. A low level ICV can be reanalyzed only once prior to corrective action.

8.12 A CCV (Continuing Calibration Verification Standard) is analyzed after at least every tenth sample and at the end of the sample run. Recovery limits for the CCV are ±10% and in the case of failure for a particular element, all samples following the last acceptable CCV must be reanalyzed for that element. A CCV can be reanalyzed only once prior to corrective action.

8.13 The ICB is analyzed after the ICV and is of the same source as the calibration blank. The CCB (Continuing Calibration Blank) is analyzed after each CCV. Concentrations of any analytes detected must not exceed the MRL or corrective action, such as a single reanalysis and evaluation, must be taken. If the ICB/CCB still fails, reanalysis of all samples since the last valid ICB/CCB for that element is required.

DoD QSM Version 3: The acceptable absolute value of the ICB and CCB must be < 2x MDL. DoD QSM Version 4.1: The acceptable absolute value of the ICB and CCB must be < LOD.

8.14 The ICS (ICSA and ICSAB) standards must be run at the beginning, every 8 hours and at the end of each analytical run.

8.14.1 The ICSA solution, containing the Al, Ca and Mg at 500mg/L, and Fe at 200mg/L, must be run at all wavelengths used for each analyte reported. The analytical results for those target analytes with MRLs ≤ 10ug/L shall fall within ± 2x MRL of the analyte's true value (the true value shall be zero unless otherwise stated). If the results for these analytes fall outside the ± 2x MRL window, check that the background correction factors are appropriate, and readjust if necessary.

Recalibration may be necessary. For analytes with a MRL > 10 ug/L, the ICSA results shall fall with ± one MRL of the analytes true value (0).

DoD QSM V3: the absolute value of all non-spiked analytes < 2x MDL, DoD QSM V4.1: the absolute value of all non-spiked analytes < LOD.

8.14.2 The ICSAB contains Al, Ca, Mg and Fe at the same concentrations as the ICSA as well as all other analytes of interest. Recovery limits for the ICSAB must be within ± 20% of the true value. If the ICSAB recoveries do not meet acceptance criteria, corrective action must be taken. Check that the background correction factors are appropriate, and readjust if necessary. Recalibration may be necessary. If the ICSAB at the end of an analytical sequence fails for a particular element, all samples must be reanalyzed for that element.

8.15 After completion of the initial requirements of this method, samples should be analyzed in the same operational manner used in the calibration routine. A 60 second rinse (rinse solution: 1% HNO_3 and 1.5% HCl) is conducted between all sample solutions, quality control samples, method blanks, and standards.

Analytical Sequence: The following QC protocol should be employed.

1. Standard S0
2. Standard S1
3. Standard S2
4. Standard S3
5. mid-range ICV(second source)
6. low-level ICV (either source)
7. ICB
8. ICSA
9. ICSAB
10. Sample 1
11. *
12. *
13. *
14. *
15. *
16. *
17. Sample 8
18. CCV
19. CCB
20. Sample 9
21. *
22. *
23. *
24. *

25. Sample 16
26. ICSA
27. ICSAB
28. CCV
29. CCB

Note: The ICSA and ICSAB sample count as analytical samples between CCV/CCBs.

Any deviations must be approved by the Inorganic Laboratory Manager or the Operations Manager before they can be implemented.

All analyses are documented in the Instrument Run Log (Attachment **3**).

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/Kg dry weight for solid samples. Results are reported to two or three significant figures.

9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with allowance for sample dilution. Do not report analyte concentrations below the reporting limit (RL) unless specifically requested by the client.

9.3 Soil concentrations are calculated using the equation below:

$$\text{Sample Conc. (mg/Kg)} = \frac{C \times V \times Df}{W}$$

Where:
C = Concentration in extract (µg/L)
V = Volume of extract (L, 100mL = 0.1L)
Df = Dilution factor (undiluted =1)
W= Dry weight of sample aliquot extracted (g)

9.4 Recovery Calculations:

The recovery of a spiked analyte is calculated as follows:

$$\text{\% Recovery (\%R)} = 100 \times (SSR-SR)/(SA)$$

Where:
SSR = spiked sample result
SR = sample concentration
SA = spike added

9.5 Relative Percent Difference Calculations:

The relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{D1 - D2}{(D1 + D2)/2} \times 100\%$$

Where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value (replicate)

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Method blanks - A preparation blank is prepped and analyzed with every batch not to exceed 20 samples. Method blank concentration must be less than or equal to reporting limits unless the sample concentration is at least 10 times greater than the blank concentration or less than reporting limits. Corrective action for method blank contamination involves determining the source of the contamination and re-prepping the affected samples in the batch. The analyst may rerun the method blank once as the first step of corrective action.

DoD QSM -Method blank concentrations must be less than or equal to one-half the reporting limit. For common contamination analytes, method blank concentration must be less than or equal the reporting limit.

10.3 Calibration verification –

10.3.1 A mid-range ICV analyzed immediately after standards must be within ±10% of the true value. The ICV is an independent source standard purchased from a different vendor than the calibration standards.

10.3.1.1 Method 6010C recommends an additional calibration verification using a low-level ICV with 70-130% criteria, however only the mid-range ICV is required to be from an independent source. In lieu of a second analysis, the response from the S3 standard can be entered back into the curve for verification.

10.3.2 The CCV is analyzed a minimum of every 10 samples in the analysis and at the end of the analytical sequence. If the closing CCV does not meet the criteria, the CCV and all analyses from the opening CCV must be re-analyzed after the problem has been eliminated.

10.3.3 The ICV and CCV analyses are followed by the ICB and CCB analyses, respectively. The ICV and CCV must pass the ±10% criteria or be re-analyzed.

10.4 Use of all standards made from a primary standard must not exceed the primary standard's expiration date.

10.5 Matrix spike (MS) samples - A matrix spike is processed with each batch of samples. Spike recoveries must be within 75-125% of the expected value. If the native sample results exceed 4x the spike added, no further action is needed. Note in narrative. Unless superceded by project requirements, it is not necessary to spike Na, K, Ca, and Mg for waters; or Na, K, Ca, Mg, Fe, Al and Mn for soil.

DoD QSM: Spike recoveries must be within 80-120%. Precision requirements are ≤ 20 % RPD for both aqueous and soil matrices. There is no corrective action for MS recoveries outside the acceptance range other than data qualification.

10.6 Matrix duplicate (DUP) or matrix spike duplicate (MSD) samples (Both options are allowed in method SW6010) - Duplicates are prepped and analyzed with every batch not to exceed 20 samples. Relative Percent Difference (RPD) is calculated for the results of duplicate samples.

10.6.1 A limit of 20% RPD shall be used for sample values greater than 5x the MRL

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10.6.2 A control limit of (±) the MRL level must be used if either the sample or the duplicate value is less than 5x MRL.

10.6.3 If one result is above 5x MRL and one below, use the ± MRL criteria.

10.6.4 If both values are below the MRL, no RPD is calculated.

DoD QSM- DUP precision is evaluated for all analytes. Precision requirements are ≤ 20 % RPD for both water and soil. There is no corrective action for MS/MD precision outside the acceptance range other than data qualification.

10.7 Laboratory Control Sample (LCS) - is prepped with a minimum of every 20 samples of the same matrix.

10.7.1 For an aqueous LCS sample, mixed standards are spiked into a beaker of DI water resulting in concentrations approximately ½ the concentration of the high calibration standard and prepped as an aqueous sample. Recoveries must be within the established control limits. The ID of the aqueous LCS sample is LCSW.

10.7.2 For soils, approximately 1g of Teflon Chips (Chemware Ultra-pure PTFE boiling stones, acid washed) may be used to simulate solid matrix is spiked with

standards at the approximate mid-point of the calibration curve. The LCS is then prepped as a soil. The ID for the solid LCS sample is LCSS.

DoD QSM –The acceptance range for both water and soil is 80-120% except silver which has a 75-120% range for soils. When Mitkem in-house LCS limit fall within DoD limit, in-house limits may be reported. If Mitkem in-house limits are wider than DoD limits, the 80-120 % limits must be used.

Laboratory control sample acceptance limits are based on control charts, which are established at Mitkem. These are referred to as in-house limits. Mitkem may also adopt limits from outside agencies when necessary.

10.8 Post digestion spike (PDS) addition - An analyte spike added to a portion of the sample digestate. A PDS is analyzed with each preparation batch of samples (PDS spiking of the same sample as used for matrix spiking is recommended) and is suggested when a new or unusual matrix is encountered within a batch. The acceptance criteria for PDS are 80-120%. The PDS is spiked at the same concentrations as the matrix spike. The PDS concentration should be >10 times the MRL, but < 100 times. If the PDS is not recovered within the specified limits, a matrix effect is confirmed.

DoD QSM V4.1: A PDS is required when the serial dilution test fails. In addition, a PDS is recommended for projects where all samples are below 50X LOD. The acceptance window is 75-125%.

Corrective action should be taken if the spike concentration is at least 2x the native sample concentration:

(a) If the MS fails for any analyte but the PDS passes, the sample may be redigested and analyzed, except when low MS recovery can be explained as a historically poor performer (example: Antimony (Sb) for soils) in that matrix. In these instances the analyst may want to discuss the outliers with the project manager or department supervisor. When redigestion is warranted, and the MS fails in the redigestate, a matrix issue is confirmed. If the MS passes for the redigestate, the initial results were due to human error during digestion or spiking.

(b) If both the MS and PDS fail, the laboratory will perform a dilution test, or may make a reasonable effort to address the matrix interference by performing the method of standard additions (MSA) or internal standard addition.

10.9 Dilution test (Serial Dilution) – should be performed with each preparation batch. If the analyte concentration is sufficiently high (minimally, a factor of 10 above the MDL), an analysis of a 5X dilution should agree within ±10% of the original determination. If not, a chemical or physical interference effect is confirmed. A serial dilution should be run whenever the PDS fails, to confirm matrix effect.

DoD QSM: A Serial Dilution is required once per preparation batch. If the SD fails, a PDS must be run.

As a general rule, Mitkem performs a PDS and SD with each preparation batch of samples.

10.10 Instrument detection limits (IDLs) are established at the time the instrument is set up and every six months thereafter. Ten solutions of diluted acid (1% HNO3 and 1.5% HCl) are analyzed on three non-consecutive days. The IDL for a particular element is three times the average standard deviation of the measured concentration for that element. An IDL is valid in the condition that IDL on both instruments are less than MDL currently reported.

DoD QSM: IDLs are established at the time the instrument is set up and whenever significant changes are made. IDLs must be < LOD.

10.11 Method detection limits (MDLs) may be established once a year. Mandatory MDL determination has been removed from the SW846 6010C method however until regulatory agencies are in agreement, Mitkem will continue to perform MDL studies. The MDL is obtained by multiplying the standard deviation of seven analyses by the appropriate one-sided 99% t-statistic. The value of this statistic equals 3.143 if the number of analyses is seven. The concentration of the analyte in the analyzed solution should be between three to five times the calculated MDL. An MDL verification check is performed immediately following the MDL study. The MDL verification check sample is spiked at approximately 1-4 times the current MDL and prepared as if it were a sample. The MDL verification check is used to establish the DoD LOD. The LOD is verified quarterly on all instruments by matrix.

10.12 Sample Dilutions: When analytes exceed the established linear dynamic range, the sample must be diluted sufficiently to bring those elements into the linear range. The dilution factor (Df) must be integrated into the final concentration calculation.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and sample results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the supervisor. The laboratory manager reviews 100% of the data prior to report generation. The QA Director or designee randomly reviews 10% of the data reported by the laboratory. Refer to Section 11 of the QAP for details.

11.2 Electronic files are validated by the analyst and transferred to the report generation group via the LIMS. Once data is in LIMS it is reviewed and validated again by the supervisor or his/her designee. The data are then locked and forms may be generated.

15. References

Fassel, V.A. et al. Simultaneous Determination of wear metals in lubricating oils by Inductively-Coupled Plasma Atomic Emission Spectrometry . Anal. Chem. 48: 516-519, 1976.

Patel, B.K.; Raab, G.A.; et al. Report on a single laboratory evaluation of Inductively coupled optical emission Method 6010; EPA contract No. 68-03-3050, December 1984.

U.S. Environmental Protection Agency. I*nductively Coupled Plasma Atomic Emission Spectroscopy* Method 6010C, SW-846 test methods for evaluating solids waste, Revision 3, Final Update IV, February 2007.

U.S. Environmental Protection Agency. Inductively Coupled Plasma-Atomic Emission Spectrometry Method for the Analysis of Waters and solids, EMMC, July 1992.

Fassel, V.A. et al. Simultaneous Determination of wear metals in lubricating oils by Inductively-Coupled Plasma Atomic Emission Spectrometry . Anal. Chem. 48: 516-519, 1976.

Winge, R.K. et.al. Inductively Coupled Plasma- Atomic Emission Spectroscopy: An Atlas of spectral Information, Physical Science Data 20. Elsevier Science Publishing, New York,1985.

Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006, and Version 4.1 April 2009.

Attachments:

1. **Attachment 1**: Analytes of Interest (Table 1 from SW-846 6010C)
2. **Attachment 2:** DoD QC Requirements, Versions 3 and 4.1.
3. **Attachment 3**: Standard Preparation Logs
4. **Attachment 4**: Instrument Run Logbook

11.3 Reports are generated by the reporting group in conjunction with the metals lab. The data submitted for report preparation is dependent on project requirements.

12. Corrective Action Procedures

12.1 Corrective action to be implemented in the event QC results are outside of the acceptance range are covered in **Section 10**.

12.2 Corrective action reports (CARs) are initiated through the LIMS in the event of an out-of -control situation that cannot be corrected by the analyst. The procedure for initiating a CAR for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel on the bookcase between the GC/MS and OPrep labs. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan, and have read the Mitkem Contingency Plan.

In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

13.2 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.

13.3 Many metal salts are extremely toxic if inhaled or swallowed. Use good housekeeping practices in areas where metal salts are being used and wash hands thoroughly after handling.

13.4 Inductively coupled plasma sources emit radio frequency radiation and intense UV radiation.

13.5 Basic good housekeeping practices such as the wiping up of spills immediately and regular cleaning of counters and hoods will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's Quality Assurance Plan.

Attachment 1
Analytes of Interest

TABLE 1

RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Element	Wavelength[a] (nm)	Estimated IDL[b] (µg/L)
Aluminum	308.215	30
Antimony	206.833	21
Arsenic	193.696	35
Barium	455.403	0.87
Beryllium	313.042	0.18
Boron	249.678 x2	3.8
Cadmium	226.502	2.3
Calcium	317.933	6.7
Chromium	267.716	4.7
Cobalt	228.616	4.7
Copper	324.754	3.6
Iron	259.940	4.1
Lead	220.353	28
Lithium	670.784	2.8
Magnesium	279.079	20
Manganese	257.610	0.93
Mercury	194.227 x2	17
Molybdenum	202.030	5.3
Nickel	231.604 x2	10
Phosphorus	213.618	51
Potassium	766.491	See note c
Selenium	196.026	50
Silica (SiO_2)	251.611	17
Silver	328.068	4.7
Sodium	588.995	19
Strontium	407.771	0.28
Thallium	190.864	27
Tin	189.980 x2	17
Titanium	334.941	5.0
Vanadium	292.402	5.0
Zinc	213.856 x2	1.2

TABLE 1
(continued)

[a] The wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity. Other wavelengths may be substituted (e.g., in the case of an interference) if they provide the needed sensitivity and are treated with the same corrective techniques for spectral interference.

[b] The estimated instrumental detection limits shown are provided for illustrative purposes only. Each laboratory must determine IDLs and MDLs, as necessary, for their specific application of the method. These IDLs represent radial plasma data and axial plasma IDLs may be lower.

[c] Highly dependent on operating conditions and plasma position.

Attachment 2:
DoD QC Requirements

APPENDIX DOD-B – QUALITY CONTROL REQUIREMENTS

The quality control (QC) protocols specified by the method shall be followed. In some cases the method may be ambiguous or provide insufficient detail. The specific manner in which methods commonly used by DoD should be implemented is detailed in the following tables. Modifications to the following requirements need project-specific approval by DoD personnel.

The tables describe specific quality assurance and quality control requirements for analytical methods (SW-846) commonly used when investigating DoD sites. The tables specify the method requirements, when available, as well as additional clarification and/or requirements from DoD. If possible, the actual requirement from the method is listed, although in some cases the description in the method is so lengthy that only a reference to the appropriate section is made. The methods should always be referenced, however, for clarification purposes. DoD has done its best to interpret the methods, providing clarification where there are inconsistencies between existing guidance documents, and stating DoD preferences when multiple options are acceptable. If there is a contradiction between the method and the following tables, the requirements specified in the tables shall be followed.

SW-846 Methods

This appendix is based on the method versions current at the time of publication, regardless of status (promulgated, draft, proposed, etc.). As methods are revised, subsequent versions of this manual may incorporate the changes. If the requirements in this appendix do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

Table B-1 below presents a summary of the definition, purpose, and evaluation of the major QC checks required in the subsequent QC tables (B-2 through B-10) for the various methods. The *definition* column describes generally what the QC check is and/or how it is performed. The *purpose* column describes why the check is important for assessing and measuring the quality of the data being generated. The *evaluation* column describes how to interpret the results of the QC check, particularly in the context of the results of other QC checks. This table should be used in conjunction with the instrument- and method-specific requirement tables to properly implement the methods for DoD projects. In addition, a supplementary list of acronyms and a glossary relevant to this appendix follows Table B-10.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION

QC Check	Definition	Purpose	Evaluation
Breakdown check (Endrin - Method 8081 only, DDT - Methods 8081 and 8270)	Analysis of a standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method-specified criteria, corrective action must be taken before proceeding with calibration.
Calibration blank (metals only)	Reagent water containing no analytes of interest, but acidified to the same pH as all samples.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above two times the MDL require corrective action.
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	This is a required QC procedure. All positive results must be confirmed.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C)	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-18).	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18).	NA	Samples cannot be analyzed without a valid MDL.
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change	Detection limits established shall be ≤ MDL.	NA	NA	Samples cannot be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months	Within ± 10% of expected value	NA	NA	

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration for all analytes (ICAL) (ICP: minimum one high standard and a calibration blank; GFAA: minimum three standards and a calibration blank; CVAA: minimum 5 standards and a calibration blank)	Daily initial calibration prior to sample analysis	ICP: No acceptance criteria unless more than one standard is used, in which case $r \geq 0.995$. GFAA: $r \geq 0.995$ CVAA: $r \geq 0.995$	Correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each initial calibration, prior to sample analysis	Value of second source for all analyte(s) within ± 10% of expected value (initial source)	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	ICP: within ± 10% of expected value GFAA: within ± 20% of expected value CVAA: within ± 20% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	Flagging criteria are not appropriate.	Problem must be corrected. Results may not be reported without a valid CCV.
Low-level calibration check standard (ICP only)	Daily, after one-point initial calibration	Within ±20% of expected value	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch	No analytes detected > ½ RL For common laboratory contaminants, no analytes detected > RL	Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 x MDL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < 2 x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid ICS.
LCS containing all analytes required to be reported	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. (See full explanation in Appendix DoD-D.)	If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	
Dilution test Serial dilution	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination	ICP: Perform post-digestion spike (PDS) addition GFAA: Perform recovery test CVAA: Perform matrix spike	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x MDL (ICP) or > 25 x MDL (GFAA and CVAA).
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x MDL	Recovery within 75-125% of expected result.	Run samples by method of standard additions (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition.	The spike addition should produce a level between 10 - 100 x MDL.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x MDL	Recovery within 85-115% of expected result.	Run samples by method of standard addition (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 85-115% range.	
Method of standard additions (MSA) or internal standard calibration	When matrix interference is suspected	NA	NA	NA	Document use of MSA in the case narrative.
MS	One MS per preparatory batch per matrix (see box D-15)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD or sample duplicate	One per preparatory batch per matrix	RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ	

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months.	Within ± 10% of true value.	NA.	NA.	

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes **ICP: minimum one high standard and a calibration blank;** **GFAA: minimum three standards 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, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	ICP: within ± 10% of true value; GFAA: within ± 20% of true value; CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard (ICP only)	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and greater than 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 > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	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 ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per preparatory batch.	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution test (ICP and GFAA only)	One per preparatory batch.	Five-fold dilution must agree within ± 10% of the original measurement.	ICP: Perform post-digestion spike (PDS) addition; GFAA: Perform recovery test.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 75-125% (see Table B-1).	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x LOD.	Recovery within 85-115%.	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TABLE D-18. LCS CONTROL LIMITS FOR METALS SW-846 METHODS 6010 AND 7470 WATER MATRIX[28]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	97	5	80	120	80	120
Antimony	98	4	80	120	80	120
Arsenic	98	4	80	120	80	120
Barium	99	4	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	100	4	80	120	80	120
Calcium	98	4	80	120	80	120
Chromium	100	4	80	120	80	120
Cobalt	99	3	80	120	80	120
Copper	99	3	80	120	80	120
Iron	102	4	80	120	80	120
Lead	99	4	80	120	80	120
Magnesium	98	4	80	120	80	120
Manganese	100	4	80	120	80	120
Mercury	100	5	80	120	No ME	No ME
Molybdenum	95	5	80	120	75	120
Nickel	100	4	80	120	80	120
Potassium	98	4	80	120	80	120
Selenium	98	6	80	120	75	120
Silver	97	5	80	120	75	120
Sodium	99	4	80	120	80	120
Thallium	97	4	80	120	80	120
Vanadium	99	4	80	120	80	120
Zinc	100	4	80	120	80	120

[28] The as-generated limits have been adjusted to reflect method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for method 6010, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

TABLE D-19. LCS CONTROL LIMITS FOR METALS SW-846 METHODS 6010 AND 7471 SOLID MATRIX[29]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	95	5	80	120	75	120
Antimony	96	5	80	120	75	120
Arsenic	95	4	80	120	80	120
Barium	98	3	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	97	4	80	120	80	120
Calcium	97	4	80	120	80	120
Chromium	99	5	80	120	80	120
Cobalt	98	4	80	120	80	120
Copper	97	3	80	120	80	120
Iron	100	4	80	120	80	120
Lead	95	4	80	120	80	120
Magnesium	96	3	80	120	80	120
Manganese	97	4	80	120	80	120
Mercury	100	6	80	120	No ME	No ME
Molybdenum	96	5	80	120	75	120
Nickel	97	4	80	120	80	120
Potassium	96	4	80	120	80	120
Selenium	93	4	80	120	75	120
Silver	96	7	75	120	70	125
Sodium	96	4	80	120	80	120
Thallium	94	4	80	120	80	120
Vanadium	99	3	80	120	80	120
Zinc	95	5	80	120	75	120

SOIL

[29] Some as-generated limits have been adjusted to reflect method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for method 6010, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

Table G-18. LCS Control Limits for Metals SW-846 Methods 6010 and 7470 Water Matrix[19]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	97	5	80	120	80	120
Antimony	98	4	80	120	80	120
Arsenic	98	4	80	120	80	120
Barium	99	4	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	100	4	80	120	80	120
Calcium	98	4	80	120	80	120
Chromium	100	4	80	120	80	120
Cobalt	99	3	80	120	80	120
Copper	99	3	80	120	80	120
Iron	102	4	80	120	80	120
Lead	99	4	80	120	80	120
Magnesium	98	4	80	120	80	120
Manganese	100	4	80	120	80	120
Mercury	100	5	80	120	No ME	No ME
Molybdenum	95	5	80	120	75	120
Nickel	100	4	80	120	80	120
Potassium	98	4	80	120	80	120
Selenium	98	6	80	120	75	120
Silver	97	5	80	120	75	120
Sodium	99	4	80	120	80	120
Thallium	97	4	80	120	80	120
Vanadium	99	4	80	120	80	120
Zinc	100	4	80	120	80	120

[19] The as-generated limits have been adjusted to reflect Method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for Method 6010, depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits.

Table G-19. LCS Control Limits for Metals SW-846 Methods 6010 and 7471 Solid Matrix [20]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	95	5	80	120	75	120
Antimony	96	5	80	120	75	120
Arsenic	95	4	80	120	80	120
Barium	98	3	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	97	4	80	120	80	120
Calcium	97	4	80	120	80	120
Chromium	99	5	80	120	80	120
Cobalt	98	4	80	120	80	120
Copper	97	3	80	120	80	120
Iron	100	4	80	120	80	120
Lead	95	4	80	120	80	120
Magnesium	96	3	80	120	80	120
Manganese	97	4	80	120	80	120
Mercury	100	6	80	120	No ME	No ME
Molybdenum	96	5	80	120	75	120
Nickel	97	4	80	120	80	120
Potassium	96	4	80	120	80	120
Selenium	93	4	80	120	75	120
Silver	96	7	75	120	70	125
Sodium	96	4	80	120	80	120
Thallium	94	4	80	120	80	120
Vanadium	99	3	80	120	80	120
Zinc	95	5	80	120	75	120

[20] The as-generated limits have been adjusted to reflect Method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for Method 6010, depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits.

Attachment 3:
Standard Preparation Logs

MITKEM LABORATORIES **METALS PRIMARY STD RECEIPT LOGBOOK:INSTRUMENT LAB**

DATE REC	PRIMARY STD ID	VENDOR	CATALOG#	LOT #	ANALYTE(S)	REC BY	DATE OPENED	EXPIR. DATE	MITKEM ID

100.0038-01/08

Page: _____

MITKEM LABORATORIES, A Division of Spectrum Analytical Inc.

METALS INTERMEDIATE STANDARD LOGBOOK

Prep Date	Analyte(s)	Primary/Intermediate STD ID	Initial Conc.(mg/L)	Amt. Added (ml)	Final Volume (ml)	Final Conc.(mg/L)	Diluent Used	Intermediate STD ID	Analyst	Exp. Date

Logbook ID: 100.0041 -01/08

Reviewed By: _____

MITKEM LABORATORIES **METALS WORKING STANDARD LOGBOOK**

Prep Date	Analyte(s)	Primary/Intermediate STD ID	Initial Conc.(mg/L)	Amt. Added (ml)	Final Volume (ml)	Final Conc.(mg/L)	Diluent Used	Working STD ID	Analyst	Exp. Date

Logbook ID: 100.0036 -12/07

Reviewed By: _____

Attachment 4:
Instrument Run Logbook

MITKEM LABORATORIES **SAMPLE RUN LOG: ICAP/4300DV**

Date: Analyst:

Spray Chamber: Minicone

POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID	
1			21			41			61			81		
2			22			42			62			82		
3			23			43			63			83		
4			24			44			64			84		
5			25			45			65			85		
6			26			46			66			86		
7			27			47			67			87		
8			28			48			68			88		
9			29			49			69			89		
10			30			50			70			90		
11			31			51			71			91		
12			32			52			72			92		
13			33			53			73			93		
14			34			54			74			94		
15			35			55			75			95		
16			36			56			76			96		
17			37			57			77			97		
18			38			58			78			98		
19			39			59			79			99		
20			40			60			80			100		
												101		
												102		
												103		

Comments:

Lot #s

HNO3: _____

HCL: _____

CRI: _____

Logbook 100.0129-05/08

STANDARD: _____

ICV/CCV: _____

ICSA/ICSAB: _____

Reviewed by: _____

MITKEM LABORATORIES **SAMPLE RUN LOG: ICAP/3100XL**

Date: Analyst:

Spray Chamber: Minicone

POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID	
1			21			41			61			81		
2			22			42			62			82		
3			23			43			63			83		
4			24			44			64			84		
5			25			45			65			85		
6			26			46			66			86		
7			27			47			67			87		
8			28			48			68			88		
9			29			49			69			89		
10			30			50			70			90		
11			31			51			71			91		
12			32			52			72			92		
13			33			53			73			93		
14			34			54			74			94		
15			35			55			75			95		
16			36			56			76			96		
17			37			57			77			97		
18			38			58			78			98		
19			39			59			79			99		
20			40			60			80			100		
												101		
												102		
												103		

Comments:

Lot #s

HNO3: _____

HCL: _____

CRI: _____

Logbook 00.0126-04/08

STANDARD: _____

ICV/CCV: _____

ICSA/ICSAB: _____

Reviewed by: _____

APPENDIX D

MEETING MINUTES

Technical Meeting Summary
Operable Unit 2 Pre-Design Investigation Data Quality Objectives
Portsmouth Naval Shipyard, Kittery, Maine

The following provides the summary and action items from the technical meeting held in the afternoon of December 8, 2009 at Portsmouth Naval Shipyard (PNS), Kittery, Maine to discuss the data quality objectives (DQOs) for the pre-design investigation at OU2. A follow up conference call was held on March 23, 2010 to discuss how comments generated in the December 8, 2009 meeting were to be addressed.

The following people attended the meeting and conference call:

Matt Thyng (1)	Navy PWD IR Program Manager
Linda Cole	NFECML RPM
Iver McLeod	MEDEP PM
Gail Lipfert (1)	MEDEP
Matt Audet	USEPA RPM
Debbie Cohen	Tetra Tech Facility Coordinator
Lisa Joy (1)	PWD-ME Env, Director
Tim Smith (1)	Tetra Tech Civil Engineer
Dan Witt (2)	Tetra Tech Project Manager

(1) Attended the December 8, 2009 meeting only
(2) Attended the March 23, 2010 conference call only

Comments/Decisions:

The project team reviewed the DQOs and information provided in draft versions of Worksheets 10 and 11 prepared to support DQO discussion. The following provides the major concerns and decisions related to those concerns.

Wetland Function and Value Assessment: The purpose of the assessment is to provide an understanding of the existing condition of the offshore area that could be impacted by remedial actions at OU2. If the remedial design includes activities in the offshore area, the design will need to indicate whether the activities could impact the offshore area and what actions are needed to protect the functions and value of the offshore area. A figure is needed in the SAP to show the specific area for the assessment.

CSM: Because lead, copper, and PCB contamination in the area west of the DRMO likely resulted from offloading of equipment near the entrance of the DRMO and from pushing of snow containing contaminated soil from the DRMO to the west, soil contamination pattern may be heterogeneous across the area of sampling (i.e., a series of hot spots). In addition, surface and shallow subsurface soil (top 2 to 4 feet bgs) are the most likely zone of soil to have been impacted by these operations, and underlying soil would not be expected to have been impacted if the overlying soil is not showing an impact. This should be reflected in the CSM and will affect the sampling method and phasing of selection of samples for analysis. Data for borings nearby the sampling area should be reviewed to see if the data show a vertical pattern in the data. Also, data should be reviewed to confirm why antimony is not included as a primary COC for the pre-design investigation and a statement as to why antimony is not included should be added into the DQOs.

Total PCB calculation method: Homolog analysis will be conducted and the individual homologs will be totaled to represent total PCB concentrations. For non-detected homologs, one-half the detection limit would be used for the calculations, assuming that the analysis attains the specified detection limits. The method of calculation will be included in the SAP.

Project Action Levels: The residential PRGs identified in the OU2 FS Report will be used as the Project Action Levels for the pre-design investigation to determine whether or not the site boundary and remedial areas need to be refined. If the Project Action Levels are exceeded, then the methodology for delineating remediation areas as provided in the FS Report would be used for the refinement of the remediation areas. Because this methodology relies on reviewing the spatial distribution in relation to the PRG for the different receptors, the project team will need to discuss the specific refinements to the remediation areas based on the spatial distribution of the data set. However, it is agreed that the lateral limits of OU2 would not be extended beyond the proposed sampling limits. If all of the pre-design samples for boundary refinement have soil concentrations less than residential PRGs, then further discussion among the project team would not be needed to not include the western area in the OU2 boundary. Several sampling locations are planned west of the DRMO that are within the current boundary of OU2. These locations are to assist in refinement of the remediation area in this area and will not affect the decision related to the boundary. Therefore, the results from these samples would not be used to determine whether refinement of the boundary is needed. MEDEP expressed a concern with how confidence bands and variability in the data were being accounted for in the comparison of the data to the DQO Action Levels.

The CSM and Project Action levels were discussed further on a conference call on March 23, 2010. A revised version of the CSM was presented which reflected the discussions on December 8, 2009. It was also discussed that the DQO Action levels would be expanded to include occupational and construction worker PRGs. The residential DQO Action levels would be used to determine the horizontal area where an action may be required (such as a landuse controls) and would be used to compare to the surface soil data. The more conservative of the occupational or construction worker PRG would then be used to define the vertical extent of an area that may require additional actions (such as excavation). It was discussed that the project team would address confidence in determining areas requiring an action by expanding areas which require an action where greater uncertainty exists (but not beyond the study boundary). For instance, if an area had a mix of high and low sample results (some above and some below an action level) the team could decide to account for the uncertainty in the area by including the entire area of high and low hits so that the team is confident the contamination is addressed. When the project team decides on the areas which require an action, the tendency of the team will be to remediate when there is a large exceedance of a Project action level, when there are multiple exceedances and to focus the remediation on areas where the exceedances are grouped in one area. A similar process was used to determine the areas requiring an action in the feasibility study.

Study Boundary for Soil Sampling for Maximum Extent of OU2 Boundary: The maximum extent of soil sampling for the pre-design investigation was developed based on the most likely maximum extent of where offloading or snow plowing from the DRMO would have occurred before the area was paved. A building and presence of a railroad would have prevented offloading and snow plowing along the westernmost area. The shoreline provides the southern limits. The topography provides the northern limitations. The project team agreed with the study boundary for the maximum extent from OU2 and investigation based on the available data for OU2.

Phasing of analysis of samples: The Navy presented a method where all proposed soil samples would be collected; however, analysis of the samples would be phased. There was concern that phasing the analysis based on the results of nearby grid samples could miss some areas of contamination that are not contiguous to the grids in which samples were analyzed. Larger grid spacing was discussed (approximately 50 by 50 feet instead of 25 by 25 feet); however, the proposed grid spacing would better provide the necessary information to support both boundary and remediation area refinements. Based on the CSM, impacts to soil are more likely to be seen in the upper soil column; therefore, phasing of sampling based on depth would be acceptable. Therefore, it was agreed vertical phasing of analysis

would be more appropriate than horizontal phasing. Based on vertical phasing, it was decided that all samples from 0 to 2 feet bgs would be analyzed first, followed by deeper soil analysis if the two upper samples showed contamination.

Action Items:

- Show wetlands function and value evaluation area and show where the sediment intertidal area is located.
- Add to CSM why antimony is not a primary COC.
- Include methodology for calculation of total PCBs using the homolog data (should be consistent with the methodology used in the OU2 RI/FS).
- Revise text to reflect vertical phasing of analysis of samples.
- Propose a method to evaluate the confidence of the sample data when comparing the sample results to the project action levels.